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ELECTRICAL PROPERTIES OF EPOXIES USED IN HYBRID MICROELECTRONICS

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Chapter I

Introduction to Epoxies

An epoxy group (epoxide to be exact) is a chemical compound in which an oxygen atom is joined to two carbon atoms which are united in some other way to form a bridge. The simplest compound, a three-membered ring, is highly reactive to both bases and acids (Fig. 1.1).[1]

An epoxy resin is any molecule containing more than one three-membered ring epoxy group that is capable of being converted to a useful thermoset form. These resins have a cross-linked chemical structure that is insoluble and infusible when reacted with catalysts to form epoxy adhesives.

Epoxies must exhibit certain basic characteristics in order to be useful. They must have the following properties:[2]

1. High adhesive and cohesive properties.

2. High temperature resistance.

3. Be stable in any environment.

4. Be chemically stable.

5. Be easily modified basically.

6. Have a long pot and shelf life.

7. Have a reasonable cure temperature.

8. Have mixture reproducibility.

9. Have good electrical and mechanical properties.

Since epoxies cannot be expected to meet the highest standards of all these properties, compomises are made by sacrificing quality
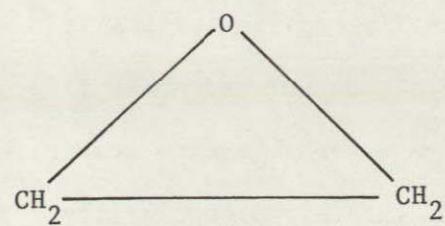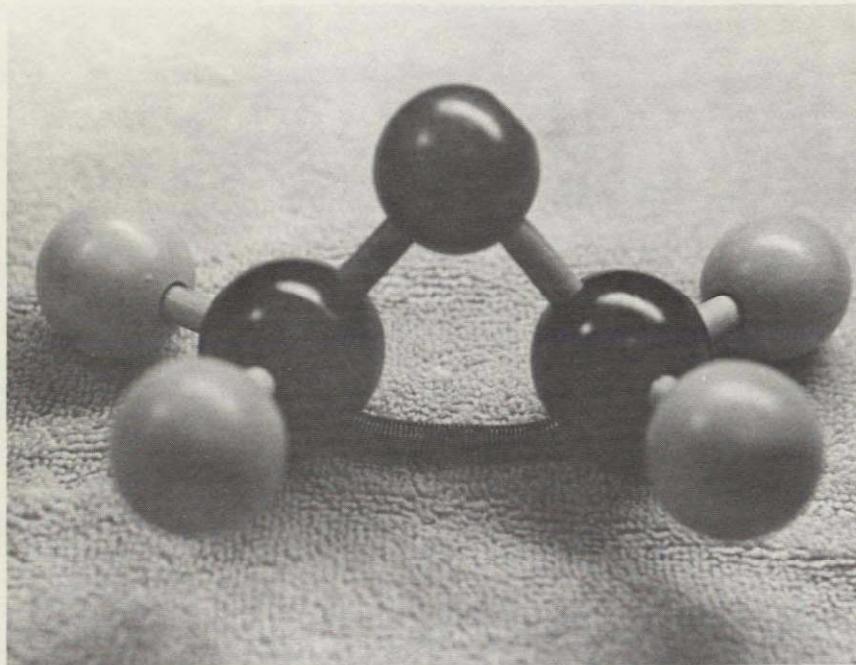
A Three-Membered Ring

Figure 1.1

in some properties while maintaining the ultimate in others. The many combinations yield an epoxy that is suitable for almost any desired use.

Epoxies have been widely used since World War II, but more recently they have been used as electrical conductors and insulators in space programs and as reinforcements on aircraft. Some of the more common applications are:[3]

    1. Adhesives.

    2. Body solders and calking compounds.

    3. Casting compounds for molds.

    4. Laminating epoxies.

    5. Potting and sealing compounds.

    6. Metal filled compounds for electrical conductors.

    7. Glass filled compounds for electrical insulators.

In the next few chapters consideration will be given only to those epoxies used in hybrid microelectronics. The most commonly used resins, catalysts, and fillers along with their effect on the chemical, thermal, mechanical and electrical properties of epoxies will be discussed in detail. Various measurement techniques will also be considered.

Footnotes

[1] Henry Lee and Kris Neville, ed., <u>Handbook of Epoxy Resins</u> (New York, McGraw-Hill Book Company, 1967), p. 1.1.

[2] Ibid., p. 1.5.

[3] Ibid., p. 1.6.

Chapter II

Adhesive Composition

Epoxy resins are unique in that they can combine with one of the

many catalysts and/or fillers to form adhesives for almost any purpose.

Catalysts, often considered to be the most important part of the system,

react with the epoxy resins to change them from a liquid state to a

hard thermoset state without forming any by-products. These reactions

may require a considerable length of time to form the hard thermoset

state required; therefore accelerators are added to the epoxy-catalyst

mixture to speed up the curing process. Fillers generally determine

the electrical properties of epoxies, but they also aid in many of the

other property functions as well.

Three of the more common epoxy resins are the diglycidyl ethers

of bisphenol A, the novolacs, and the cycloaliphatics (Fig. 2.1).[1]

The diglycidyl ether of bisphenol A (DGEBA) is a product of the reaction

of epichlorohydrin and bisphenol A in the presence of an alkaline cat-

alyst, normally sodium hydroxide. By controlling operating conditions

and varying the ratio of bisphenol A to epichlorohydrin, the degree

of polymerization and, therefore the molecular weight, can be varied

thus yielding epoxies of varying physical properties.

Novolac epoxies are formed by reacting novolac resins with epi-

chlorohydrin in a manner similar to that of DGEBA, and when cured with

any conventional catalysts, they produce a tightly cross-linked system.

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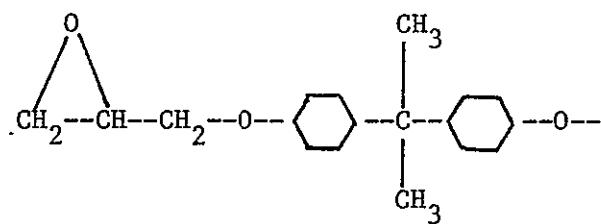
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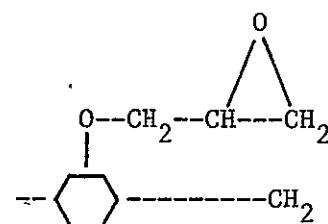
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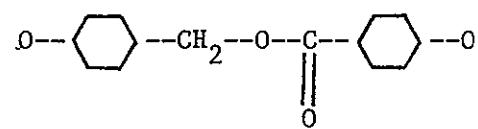
Cycloaliphatic Epoxy

Figure 2.1

Novolac resins contain a benzene ring type structure which tends to
make them exceptionally stable thermally as well as highly reactive
and versatile with all known catalysts. Because of their tightly
cross-linked structure, novolacs exhibit higher heat distortion,
better chemical resistance, and higher adhesion than the bisphenol
based epoxies.

The cycloaliphatic resins are products of the condensation of an
acid with epichlorohydrin followed by dehydrohalogenation. These resins
are distinguished by the location of their epoxy group on the ring
structure instead of on the aliphatic chain as are the novolacs. Cyclo-
aliphatic resins are superior to all others when arc-track and weather-
ing resistance are required.

Although not epoxies, polyimides and polyamide-imides are often
thought of in conjunction with epoxies and in the microelectronics
field, they have roles that are not too dissimilar from those of epoxies.
Polyimides and polyamide-imides are polymers of rigid molecules formed
by the polycondensations of aromatic acid anhydrides and aromatic
diamines. Polyimides are heterocyclic polymers having a nitrogen atom
in the imide ring instead of a carbon atom commonly found in other
resins (Fig. 2.2a). Polyamide-imides contain aromatic rings as well as
the nitrogen link of the imides (Fig. 2.2b).[2]

Catalysts or curing agents cause the short-chained polymers of
uncured resins to join together in long cross-linked structures
(Fig. 2.3), thereby controlling the end properties of the epoxies.
Aromatic and aliphatic amines, catalytic curing agents (amides), and
anhydrides are the main catalysts used with the epoxy resins mentioned
above. Aromatic amines are amines which have a nitrogen atom attached
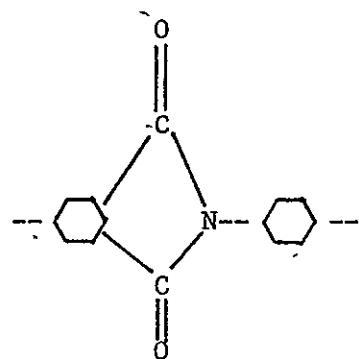
a. Polyimides
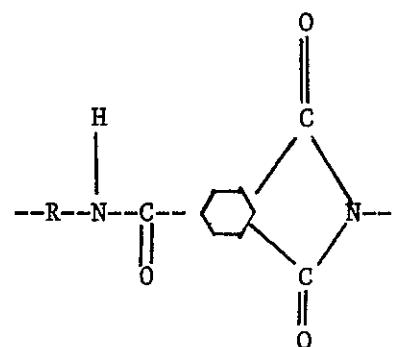
b. Polyamide-imides

Figure 2.2

Cross-linked Structure

Figure 2.3
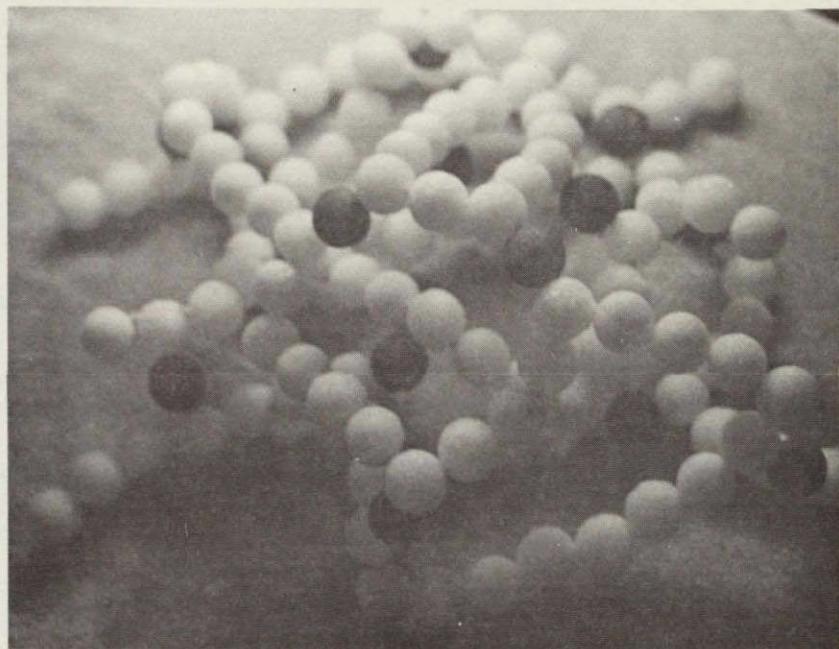
directly to an aromatic ring. These amines usually require an elevated temperature to cure and some even require low temperature pre-cures to minimize the exotherm and a high temperature post-cure to bring out the maximum properties.

Aliphatic amines are structures that contain no benzene ring except those in which the nitrogen atom is not directly tied to the benzene ring. These amines react faster than do aromatic amines due to a higher basicity. Aliphatic amines allow curing at room temperature, but due to high exothermic temperatures during cure, the amount of epoxy used at one time is limited..

Catalytic curing agents in general have a longer working life, but like the aromatic amines, require high temperature cures or two-part cures. These curing agents are seldom used alone due to their weakly reactive hydrogen atoms. Instead, two or more are often used together to provide a more functional and faster reacting compound. In many cases, these curing agents are also used with other types of catalysts to serve as accelerators.

Acid anhydrides are the second most widely used reactants for curing epoxy resins. The chemical linkages of the epoxy acid arrangement causes anhydride cured resins to be more caustic-sensitive than amine cured systems. Although they are generally very stable thermally and chemically, the low exotherms of anhydride systems make it necessary that they be cured for long periods of time at elevated temperatures. This involves risks of vaporization with some volatile anhydrides. Reactions of the anhydrides with the resin are complex and dependent on gel time and temperature, post-cure time and temperature, presence of accelerators, type of accelerators, amount of hydroxyl present,

Fillers should be neutral or slightly basic and nonreactive with either

the resins or the curing agents. For microelectronic purposes, fillers

such as glass and silica are used in nonconductive epoxies while gold

and silver are used in the conductive epoxies. Fillers tend to settle

from the resins during storage. The degree of settling depends on the

specific gravity and particle size of the filler, the type of resin used,

and the vixcosity of the compound. The finer the filler particles, the

less it will settle. Consequently, fine particles of filler are often

used with coarser particles in some compounds to prevent excessive

settling. Settling becomes more significant when long-term storage is

involved or when stored at elevated temperatures which lower the vis-

cosity of the compound. Selection of a filler depends on particle size,

shape of particle, absorption characteristics, optimum loading volume,

and final electrical characteristics. Loading volume, in turn, depends

on the upper limits of permissible viscosity, system limit, and ultimate

properties desired.

In general, epoxies used in microelectronics consist of a resin,

a curing agent, and accelerator (if needed) and a filler. One company

offers a polyimide adhesive using polyimide resins with dicyandiamide

(catalytic catalyst) as a curing agent. Dicyandiamide is a strong

catalyst; therefore accelerators are not needed in this compound. Pure

silver filler makes this a very good high temperature, electrically

conductive adhesive. This company also offers an electrically insulating

adhesive which uses a mixture of aliphatic and cycloaliphatic resins and

a catalytic curing agent. The filler is calcium carbonate, a salt.

Thus it can be seen that epoxies can vary widely, not only in

difference of resins and catalysts but also in amounts of these compounds.

Epoxies are available in both conductive and nonconductive as well as one- and two-part forms. Generally speaking, the curing agent must be mixed with the resin prior to use in two-part form, whereas in one-part form, the resin and catalyst are premixed with some sort of inhibitor present to extend pot life. In the following chapters, the effect of epoxy composition on the final cured properties will be investigated.

Footnotes

[1]Charles A. Harper, ed., <u>Handbook of Plastics and Elastomers</u>
(New York, McGraw-Hill Boo- Company, 1975), p. 1.45.

[2]Ibid., p. 1.192.

Chapter III

Curing

In Chapter II, cured adhesives were referred to frequently and, in the following chapters, the degree of cure will be shown to have significant effects on all the properties of adhesives. This chapter is designed to familiarize the reader with the actual phenomenon of curing. Curing takes place first by conversion and then by cross-linking of molecules into three-dimensional networks. These phenomena and their importance, along with various methods of increasing or decreasing curing time, will be discussed here.

The term "curing" is used to describe the process in which an epoxide and a curing agent are changed from low molecular weight materials to highly cross-linked networks consisting only of the epoxide and curing agent.[1] The curing process can take place by chemical reaction, by action of heat and catalysts, alone or in combination, with or without pressure.[2] In most cases and throughout this manuscript, curing will refer to the reaction of resin and catalyst due to herat and catalysts without pressure. Conversion, the disappearance of epoxy groups, is normally equal for all systems, whereas the properties of the cured systems will vary greatly. It has been shown[3] that hydroxyl groups increase and epoxy groups decrease as reaction takes place. These hydroxyls are then used in the final stage of curing, namely cross-linking.

When chemical links are set up between molecule chains, the material is said to be cross-linked. In thermosets, cross-linking causes the formation of one large infusible molecule instead of a long chain of separate molecules[4] (Fig. 3.1). The extent of cross-linking depends on the type and the concentration of the curing agent involved. In the early stages of curing, cross-linking is very slight and the epoxy is in a fluid or paste-like state. Cross-linking here is at points far removed from each other. However, as cure continues, cross-linking becomes more general and the epoxy takes on a more thermoset state (Fig. 3.2).

While most adhesive systems need only to cure at one temperature for maximum properties, others require a low temperature cure in conjunction with a much higher temperature post-cure. The low temperature cures promote linear polymerization while the post-cures provide the necessary cross-linking. The properties of some adhesive systems cured in this fashion are often undesirable if the temperature of the pre-cure is too high; then again they may show to be improved in small low-exotherm masses over large high-exotherm masses. Conversely, other systems will form molecules at the low pre-cure temperature which prevents cross-linking from taking place in spite of the curing time and temperature of the post-cure.

The degree of cure has come to refer to the extent in which cross-linking becomes sufficient to provide optimum properties of adhesives for a particular application. The number of effective cross-links per unit volume is known as cross-linking density. In molecules of high cross-linking density, the distances between cross-links are short which results in rigid structures, whereas, the long spaces between
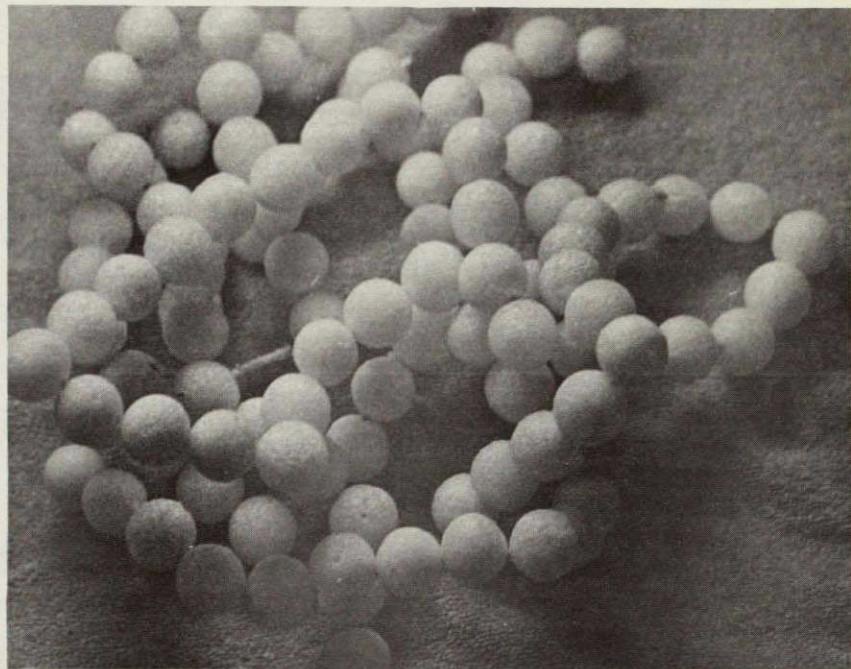
No Cross-linking
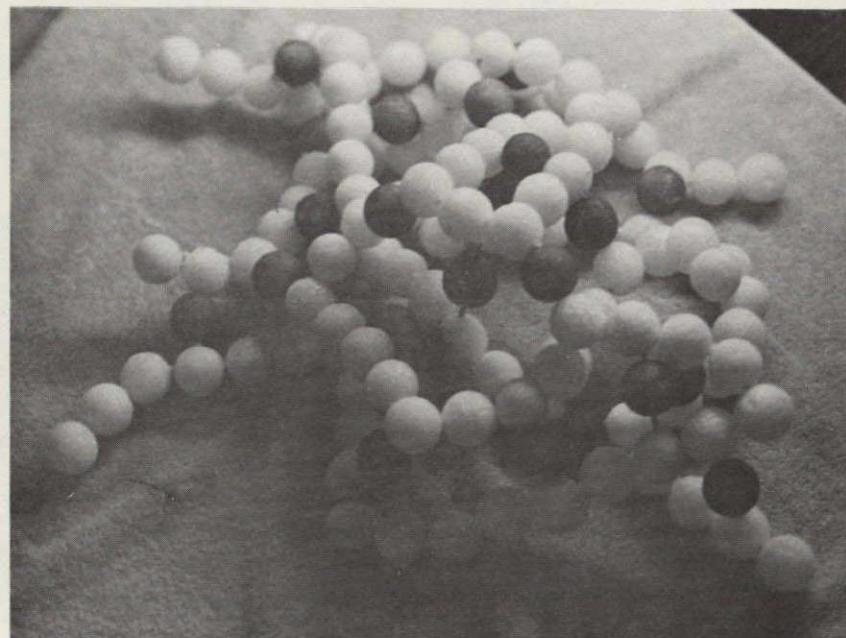
Figure 3.1

Dense Cross-linking

Figure 3.2

Footnotes

[1]Henry Lee and Kris Neville, ed., Handbook of Epoxy Resins (New York, McGraw-Hill Book Company, 1967), p. 5.1.

[2]Charles A. Harper, ed., Handbook of Plastics and Elastomers (New York, McGraw-Hill Book Company, 1975), p.4.

[3]Lee, et al., p. 1.10.

Chapter IV

Chemical Properties of Adhesives

This chapter is primarily concerned with the effects of composition
and curing on the chemical properties of adhesives. Some of the pro-
perties that will be considered are pot and shelf life, chemical resist-
ance, corrositivity, and outgassing. These properties will be discussed
in general in this chapter and in reference to the particular adhesives
used in microelectronics in Chapter IX.

To be highly chemically resistant is one of the most important
properties of adhesives used in microelectronics. Ether linked adhesives
are fairly stable against most organic and inorganic acids ad caustics,
but the carbon-amine links are even more stable, the aromatics yielding
a higher performance than the aliphatics. Ester links are also stable
against most organic acids, but prove to be very unstable when subjected
to inorganic acids and caustics. Epoxy resins are resistant to most
salt solutions and are not affected by most aqueous solutions. Anhydride-
cured systems show higher resistance to acids while amine-cured
adhesives are more resistant to bases. Due to their highly polarized
and hydrogen bonded character, adhesives are extremely sensitive to
water, resulting in a decrease in mechanical strength. Polyimides also
have a good resistance to organic and inorganic acids. They are
usually unaffected by grease and oil as well as aliphatic and aromatic
linked solvents. However exposure to concentrated acids may cause
severe brittleness of the adhesive in the cured state. Polyimides

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are attacked by strong bases such as aqueous ammonia. Oxidizing chemicals

may also cause oxidation of polyimide parts even under mild conditions.

Exposure to hot water or steam causes decreases in mechanical strength;

however, the strength may be restored by simply drying the adhesive.[1]

Fillers may affect the moisture and solvent resistance of adhesives

either adversely or advantageously. Fillers such as calcium carbonate

tend to weaken acid resistance while silica and aluminum tend to weaken

alkali resistance. Because of the failure of internal bonds, glass

fibrous fillers tend to exhibit a wicking action for moisture.

Figures 4.1 and 4.2 show the expected results of adhesives when sub-

merged in water for seven days.[2]

Users of adhesives are particularly concerned with the corrosive

effects of epoxies and polyimides on various materials. Corrosive

effects vary with adhesive systems and are usually caused by direct

chemical attack aided by the application of electric currents. Retard-

ation of corrosion may be imposed if insoluble products are formed on

the surface. Conversely, removal of insoluble products from the surface

results in an acceleration of corrosion. Perhaps the most significant

part of an adhesive system that contributes to corrosion is the catalyst.

For example, boron trifluoride (BF_3), a common catalyst, is highly

corrosive to most materials even in a diluted form. However it is still

used extensively because it cures very rapidly. Since BF_3 is most

corrosive under humid conditions, careful sealing will usually eliminate

most corrosive effects.

Outgassing under high vacuum is also of interest, especially to

space technology. Outgassing is usually measured in terms of weight

loss. Low weight losses, 10^{-6} to 10^{-7} torr, have been found to

Water Absorption after seven days immersion for DGEBA systems.

Figure 4.1

Water absorption versus filler for cured DGEBA

Figure 4.2

1. Resin
2. Clay filler
3. Calcium carbonate
4. Microsil
5. Silica
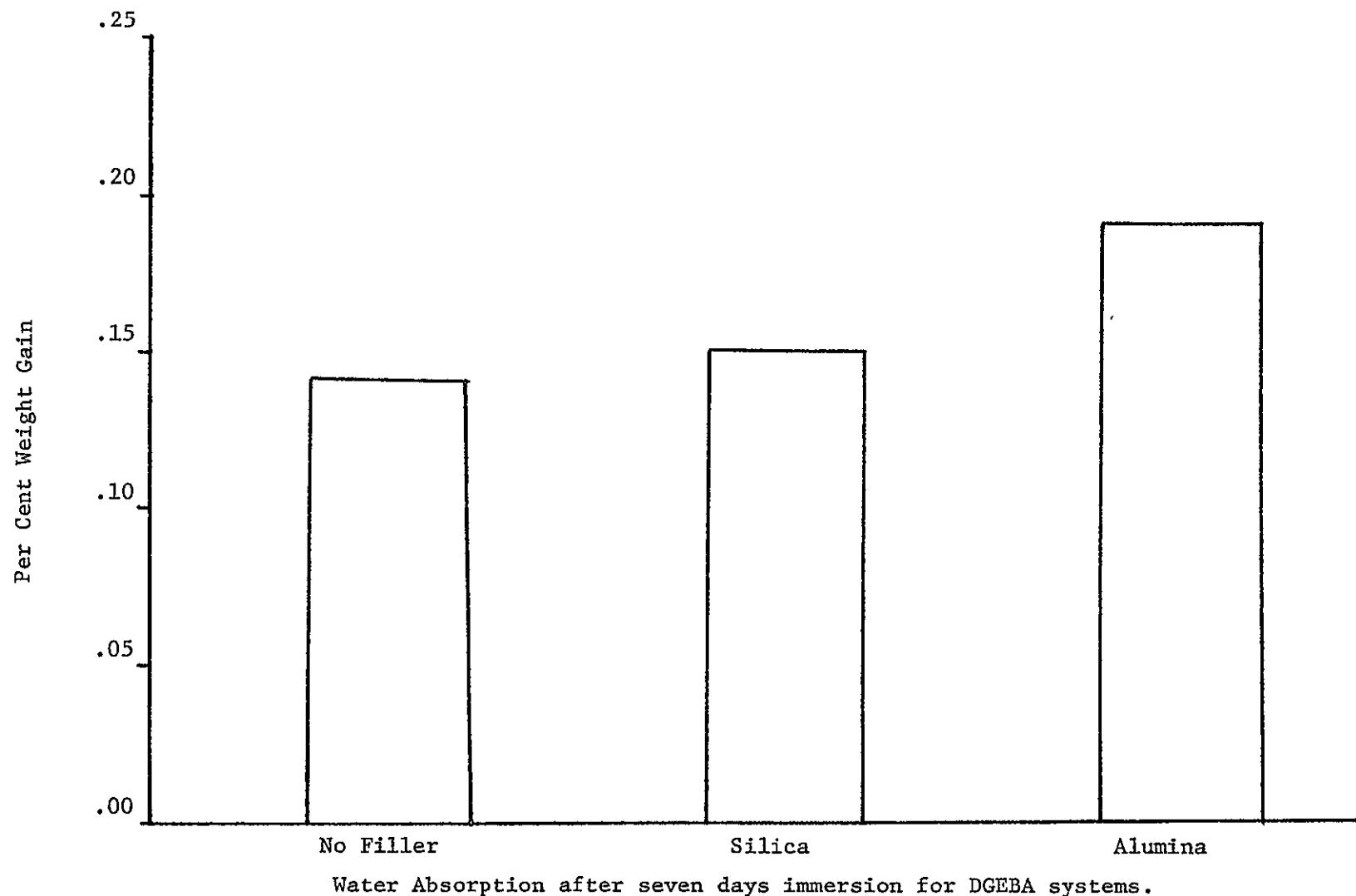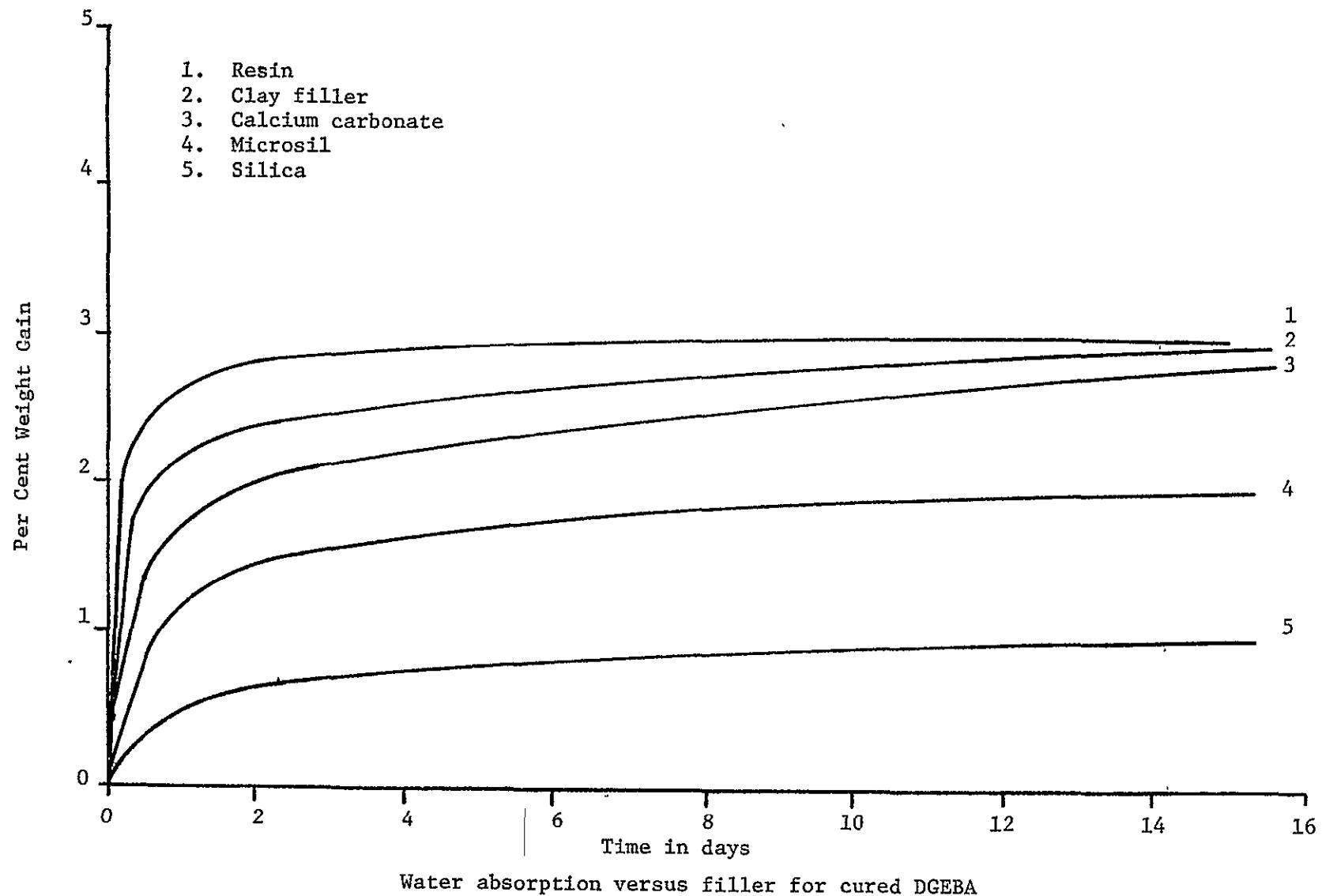
be typical of most epoxy systems. Resins cured with polyimides experience

a greater weight loss than those resins cured with anhydride catalysts.

Amine cured resins come in between these two systems in weight loss.

Figure 4.3 shows the effect of aging in a vacuum on weight loss.[4] Out-

gassing is more prevalent in systems that use modifiers such as alcohol

to decrease the viscosity of the adhesive.

Viscosity is one of the most used measurements in guiding the

flow properties of adhesives. Systems of high viscosities are usually

too thick to flow properly for encapsulating purposes; however they

may be just right for device mounting. A too low viscosity will flow

too much, thus not encapsulating fully. Viscosity may be increased by

the use of fillers or decreased by increasing its temperature. For a

given resin type, viscosity increases in proportion to the higher

molecular weight present. Aromatic-based adhesives are generally solid

or semi-solid at room temperature, but blends of these resins may result

in a liquid form. Aliphatic resins have an even lower viscosity than

the aromatics (Fig. 4.4).[5] Fillers tend to increase viscosity depending

on type and size of filler particle. Fibrous fillers increase the most,

non-fibrous increase the least. Finer particles generally increase

viscosity more than equal weights of larger sizes particles.

Pot life is the length of time an adhesive remains usable after

the resin and curing agent are mixed. In systems where reactivity

between resin and catalyst occurs at elevated temperatures, thus allow-

ing mixing of the two components long before using, pot life is

synonomous with shelf life. Shelf life is the length of time an

adhesive can retain its maximum properties before using. Both pot and
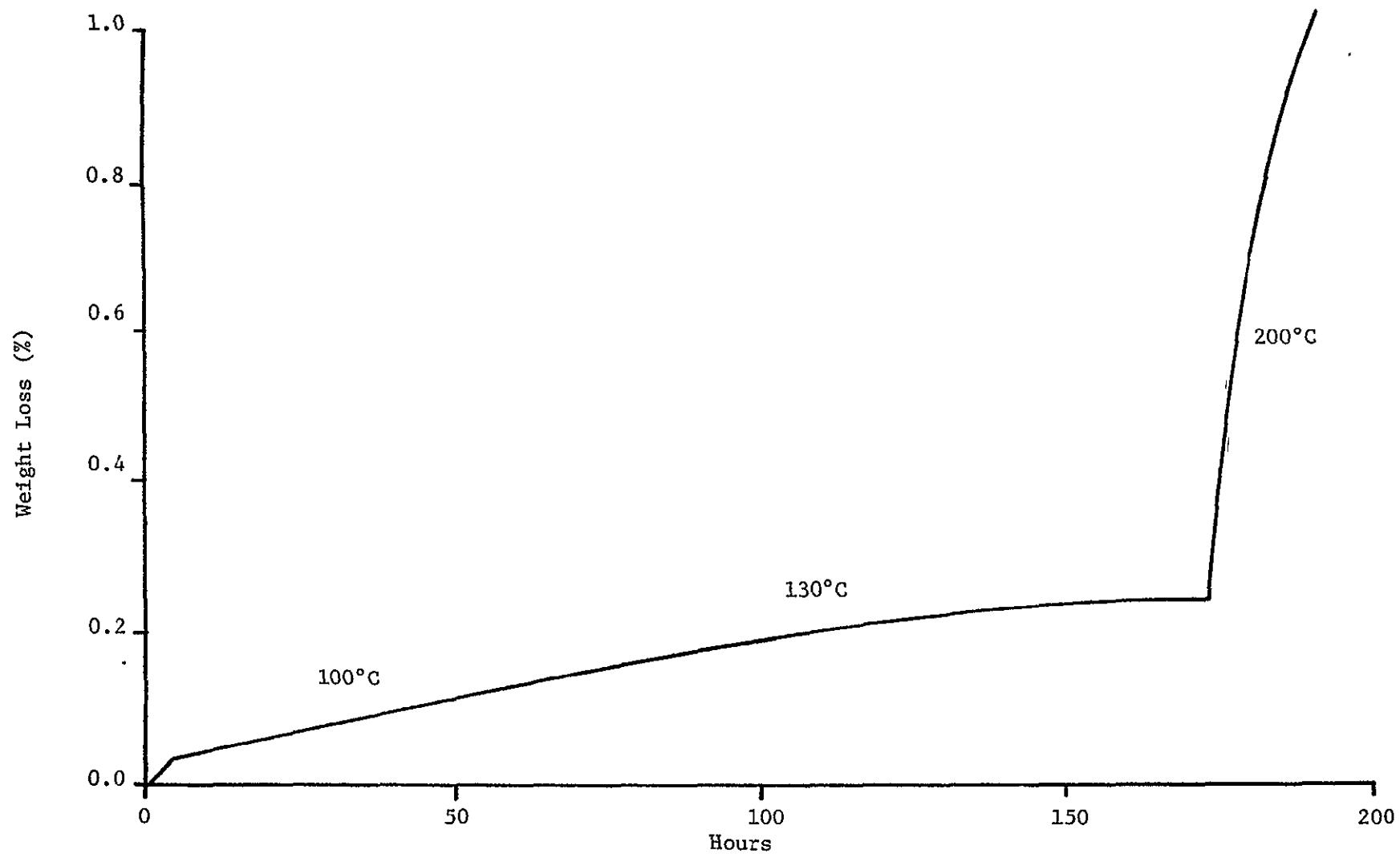
Weight loss upon vacuum aging at 10^{-6} torr at temperatures shown for DGEBA.

Figure 4.3
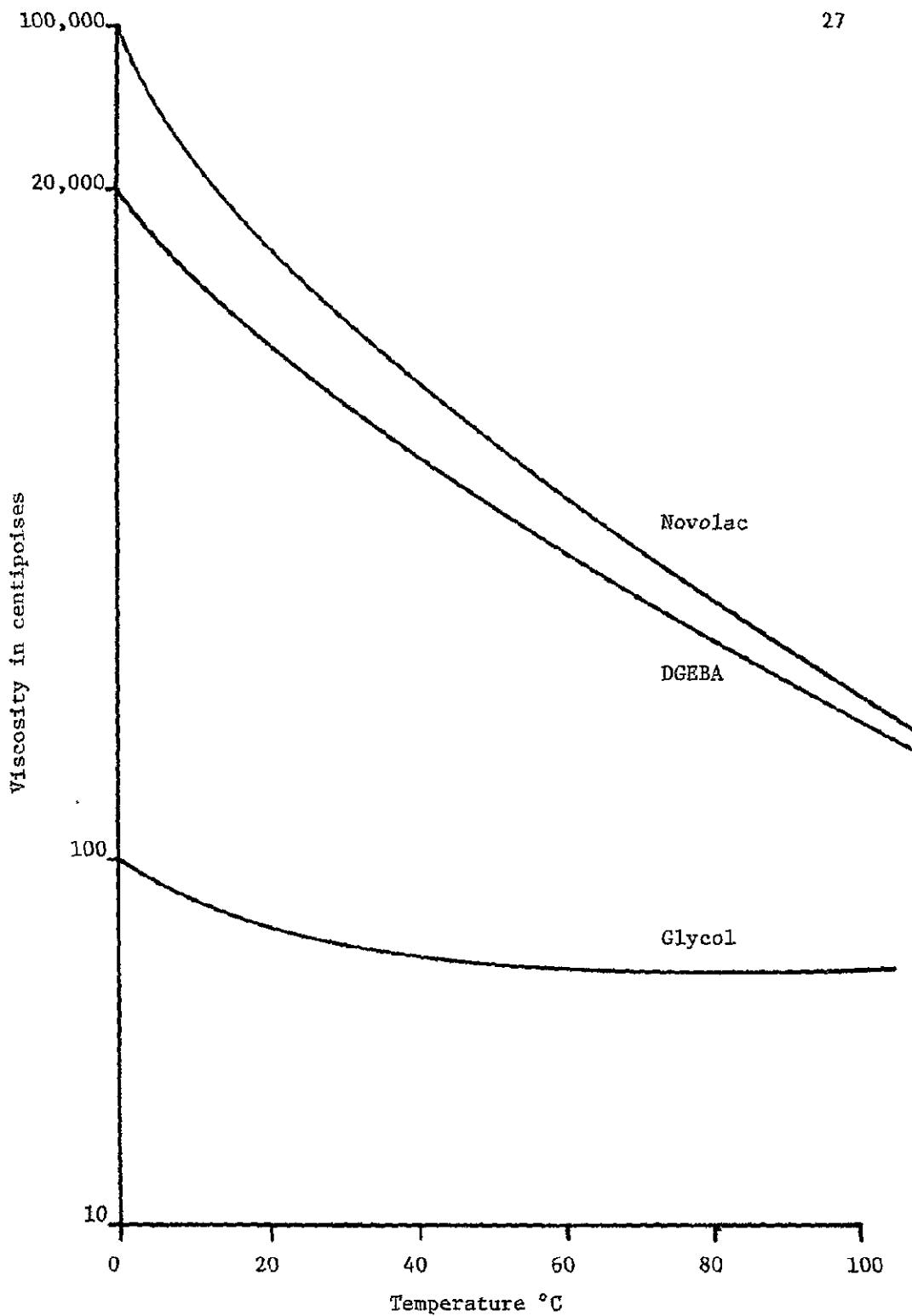
Figure 4.4

shelf life may be extended by selection of proper resins and catalysts and by storing at temperatures below 20°C.

When choosing an adhesive, several factors must be considered. For example, if contact with a metal is imminent, a corrosive adhesive would not be desirable, or, if the adhesive is to be used in the presence of chemicals, an adhesive that is resistant to both acids and bases would be the best choice. Viscosity, too, is a very important factor to consider when choosing an adhesive. This choice depends on the use of the adhesive. These points will be examined again in Chapter IX in regards to particular adhesives used in microelectronics.

Footnotes

[1]Charles A. Harper, ed., <u>Handbook of Plastics and Elastomers</u> (New York, McGraw-Hill Book Company, 1975), p. 4.28.

[2]Henry Lee and Kris Neville, ed., <u>Handbook of Epoxy Resins</u> (New York, McGraw-Hill Book Company, 1967), p. 14.43.

[3]Ibid., p. 6.33.

[4]Harper, p. 2.41.

[5]Lee, et al., p. 4.15.

Chapter V

Thermal Properties of Adhesives

Many adhesives, especially those used in space explorations, are
required to withstand gross changes in temperature with few changes in
their properties. This chapter will discuss the thermal conductivity,
the coefficient of expansion, the temperature range of use, and the
heat distortion of adhesives in general. In Chapter IX, all of these
points eill be discussed in connection with those particular adhesives
used in microelectronics.

Thermal stability is the ability of adhesives to serve at elevated
temperatures with minimum loss in properties. Thermal stability depends
on the chemical structure and bonding as well as the cross-linking density.
Studies show that thermal decomposition begins with the rearrangement
of epoxy groups and progresses to dehydration and finally to cleavage.[1]
Most resins are carbon-to-carbon bonds, however the bisphenol resins
are primarily ether linked. The presence of aromatic groups in cured
networks tend to increase thermal stability, while in the non-aromatic
groups, the cross-linking density and formation tend to affect the
stability adversely. The cyclics or ring type groups are the most stable
while the linear and the branched groups are the less stable of the
non-aromatics. With bisphenol resins, symmetry of their substituent
parts yield much improved thermal stability at elevated temperatures.
For asymmetric substituents, stability decreases with increases in the
length of the carbon chain. Ester and ether linked resins are fair in

thermal stability while the amine groups are rather unstable. Hydroxyl

groups have vulnerable points which result in degradation at high temp-

eratures. The high chlorine content in glycidyl ethers is very detri-

mental to thermal stability (Fig. 5.1).[2] Anhydride curing agents give

resins greater thermal stability than other curing agents due to a

greater cross-linking density which results in short-term endurance at

about 150°C.

For adhesives to withstand elevated temperature exposure, they

must have a high softening point and be resistant to oxidation. High

temperature adhesives are characterized by their rigid polymeric

structure, high softening point, and basic chemical group. Of all the

adhesives mentioned in Chapter II, polyimides exhibit greater endurance

under long-term service temperatures greater than 200°C. Epoxy adhesives,

on the other hand, are generally limited to less than 150°C. Certain

epoxies, however, are able to withstand temperatures greater than 250°C

for short periods of time and for longer periods of time at temperatures

just under 200°C. These adhesives are usually conglomerates of very

stable resins and high-temperature curing agents. One of the more

successful of these conglomerates is the epoxy-phenolic (a novolac) which

can withstand temperatures greater than 350°C for short periods of time

and continuous use at 200°C. Polyimides retain about 80% of their physical

properties at 250°C. They may be used at this temperature with little

loss in effeiciency and at 490°C for short periods of time. Polyimides

also perform satisfactorily at cryogenic temperatures.[3]

The change in the length of a material with temperature is

indicated by the coeffiecient of linear expansion. Thermal expansion

is a function of the resin, the catalysts, and the filler. In general,
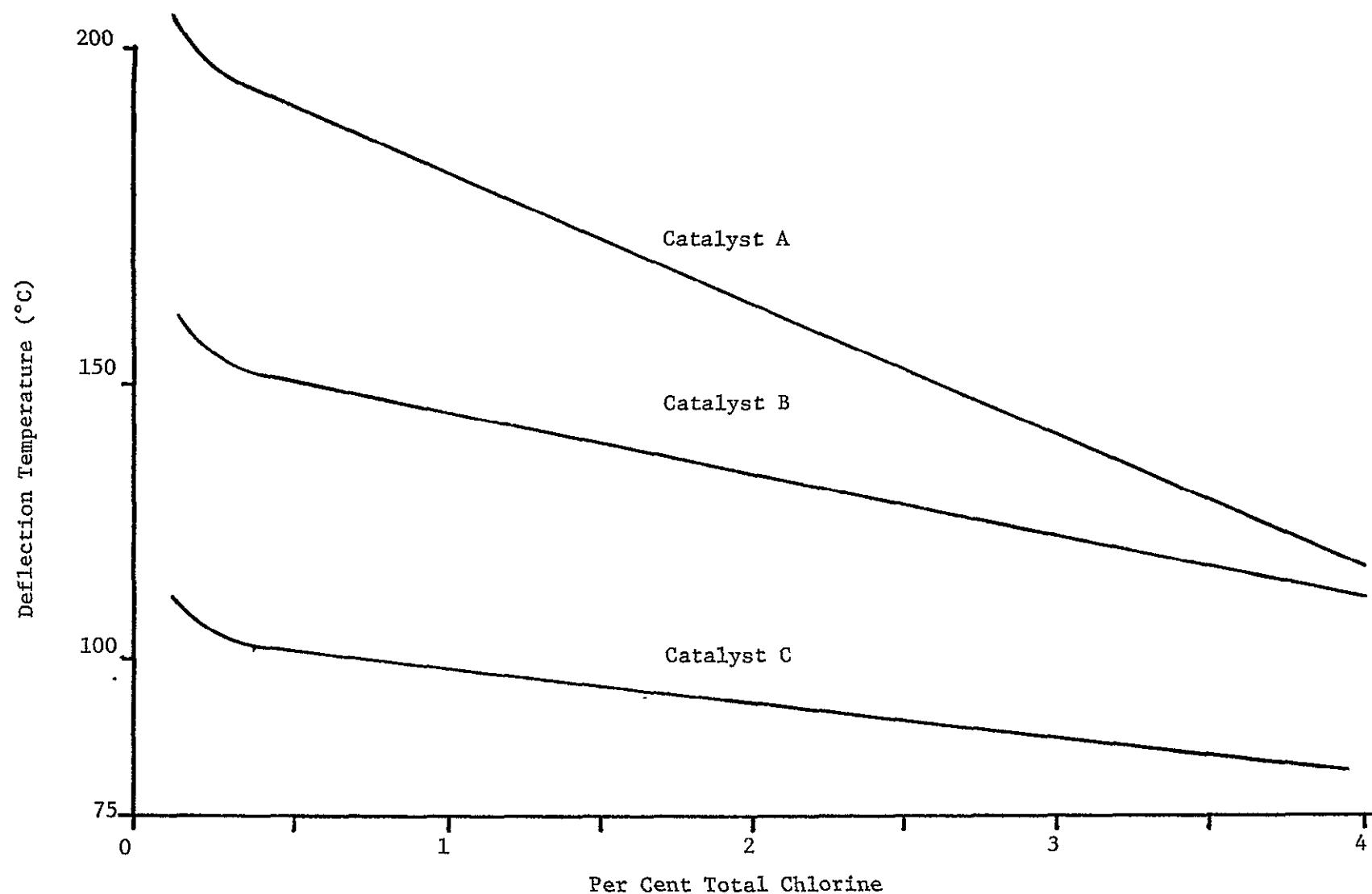
Figure 5.1

the more flexible a system the higher the expansion rate. Therefore, densely filled systems would be expected to have lower expansion rates. The resins should be matched or modified to match the fillers in expansion rate in order to avoid major stress points. Even so, microscopic stress points occur between filler particles and the resin. A comparison of the coefficient of thermal expansion between various filler materials is shown in Figure 5.2.[4] Figures 5.3 and 5.4 show the effect of hardeners and curing on thermal stress in a conventional epoxy.[5]

Thermal shock resistance can be a problem with the rigid thermosetting adhesives. Thermal shock failure generally occurs at stress points created by temperature gradients (difference in temperature between interior and surface) and external factors (e.g. centrifugal forces in rotating equipment). Some adhesives can withstand repeated cycling between 180°C and -75°C without many failures. The effect of filler on thermal shock resistance is shown in Figure 5.5.[6]

Heat distortion temperature (HDT), also known as deflection temperature, is normally 90 to 180°C for a common adhesive, but some curing agents may increase the deflection temperature to as much as 300°C. On the other hand, aliphatic cured flexible resins may show a HDT below 0°C. Cycloaliphatic resins have a high HDT but they get more brittle as the HDT increases. The effect of catalyst concentration on the deflection temperature is shown in Figure 5.6.[7] The deflection temperature point may also be improved with additional curing which, to a point, increases cross-linking. However, there is a point at which thermal degradation overcomes any improvement made by curing. Figure 5.7 and 5.8 show the effect of curing on heat distortion for typical epoxies.[8]
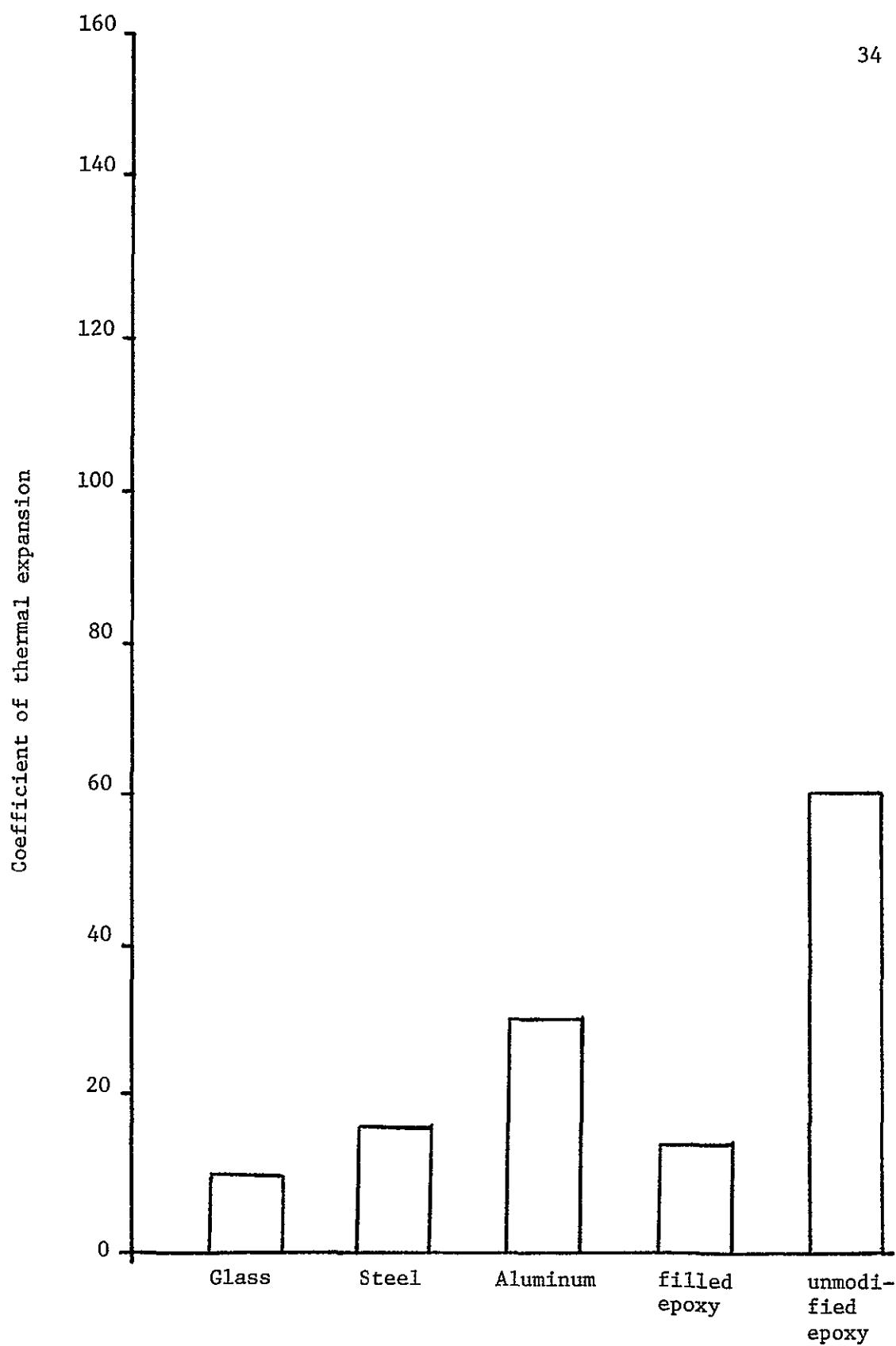
Figure 5.2
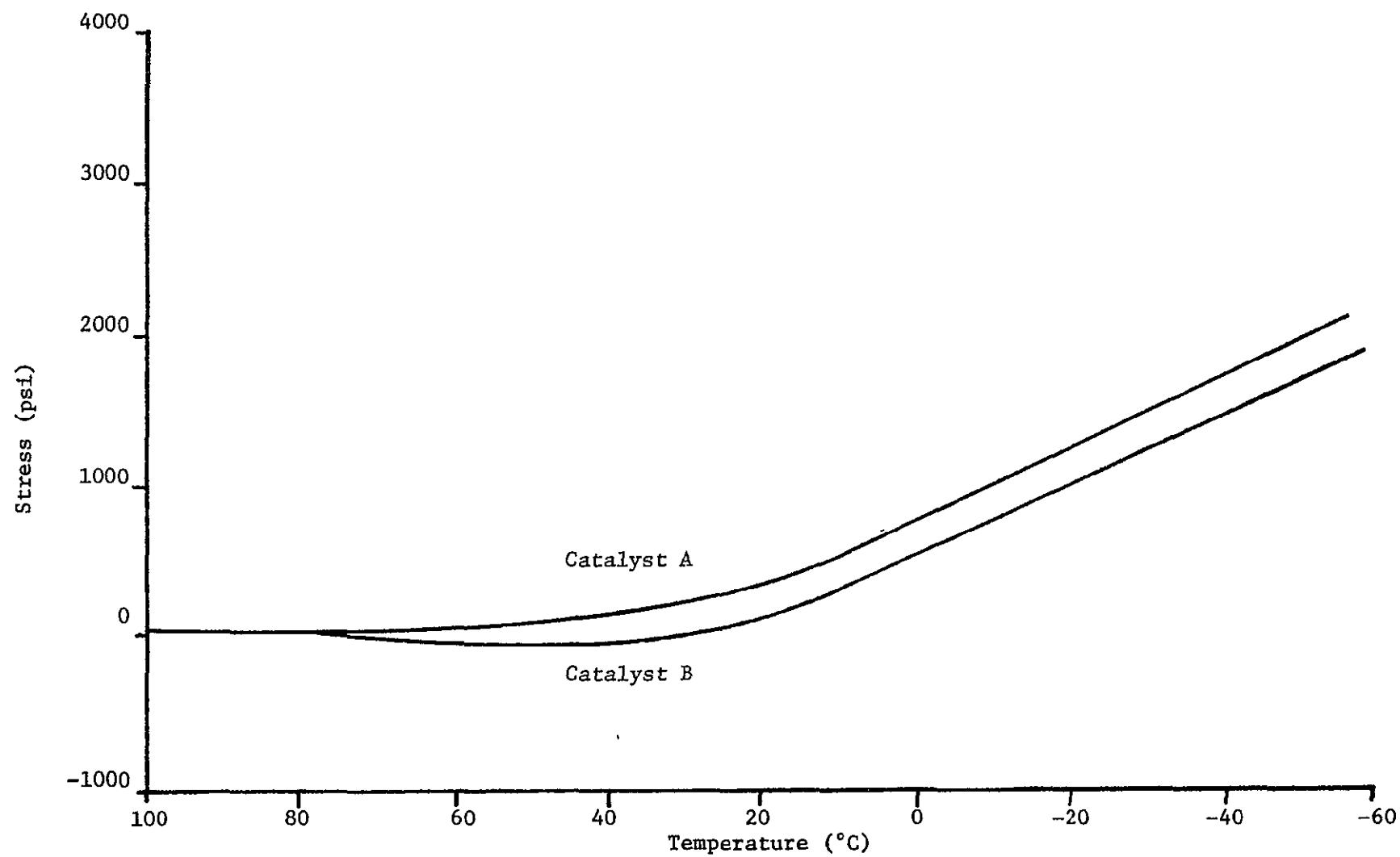
Effect of hardener on stress

Figure 5.3

Effect of curing temperature on stress

Figure 5.4

Effect of filler loading on thermal shock

Figure 5.5

Performance of epoxy under stress
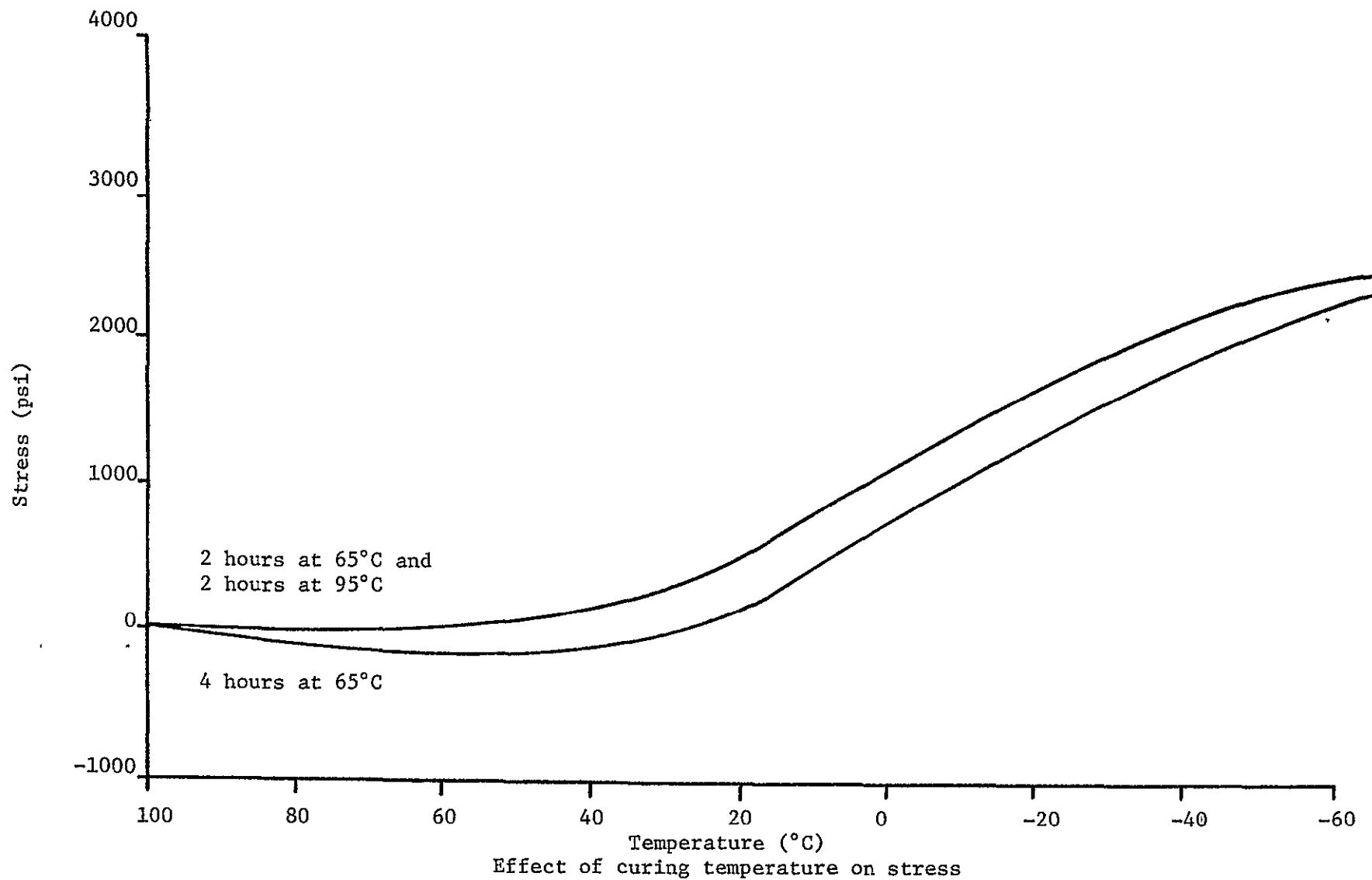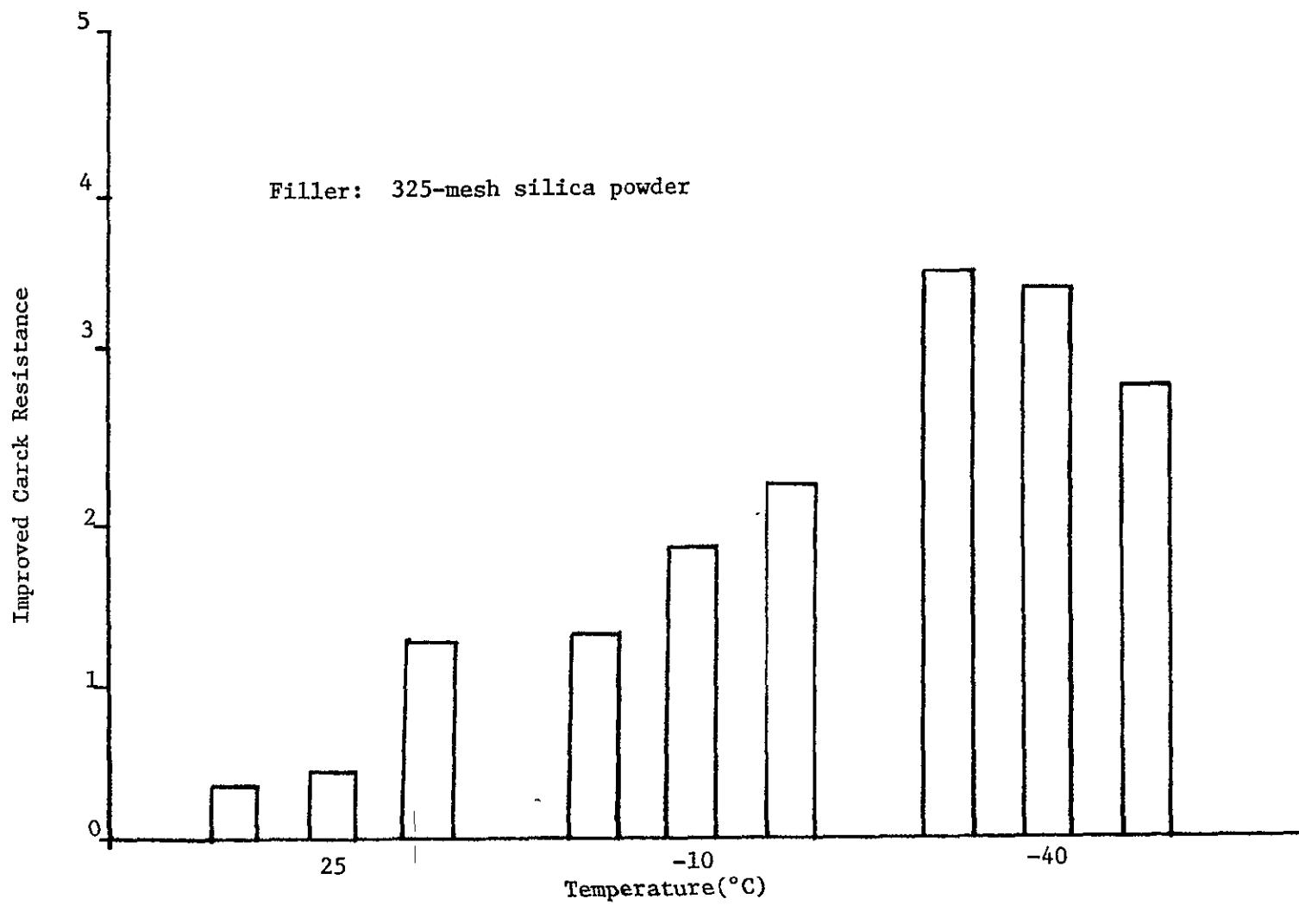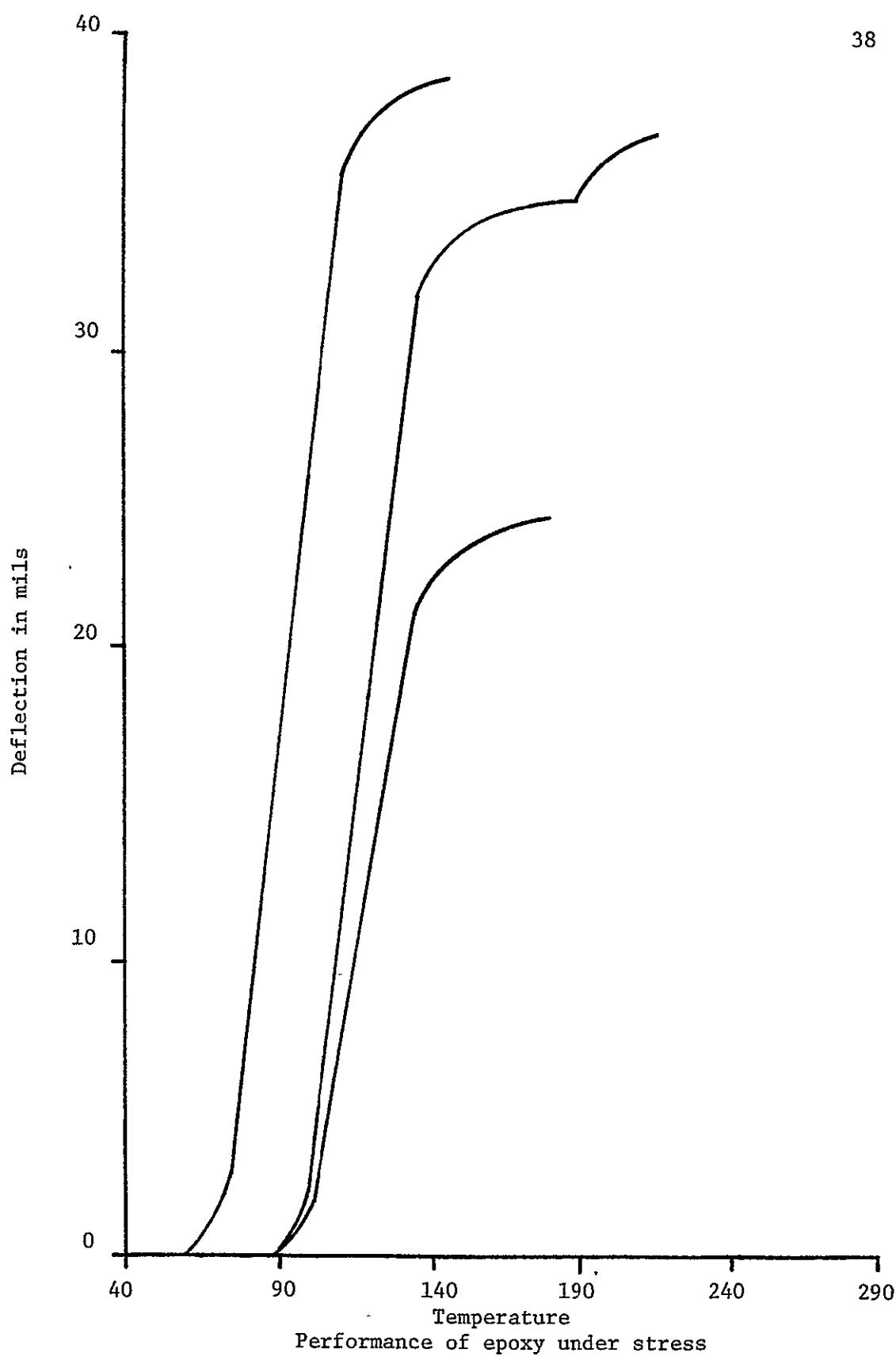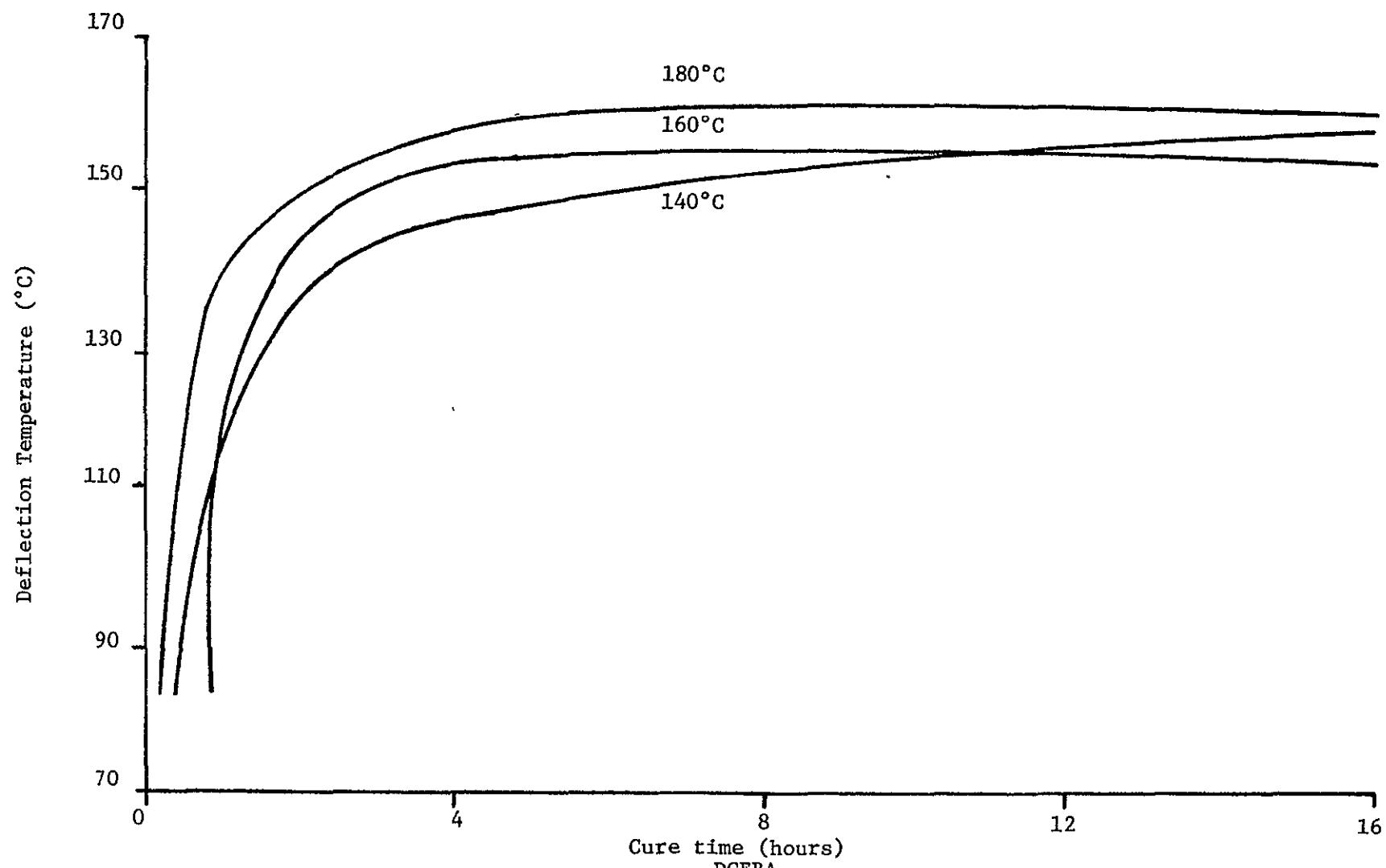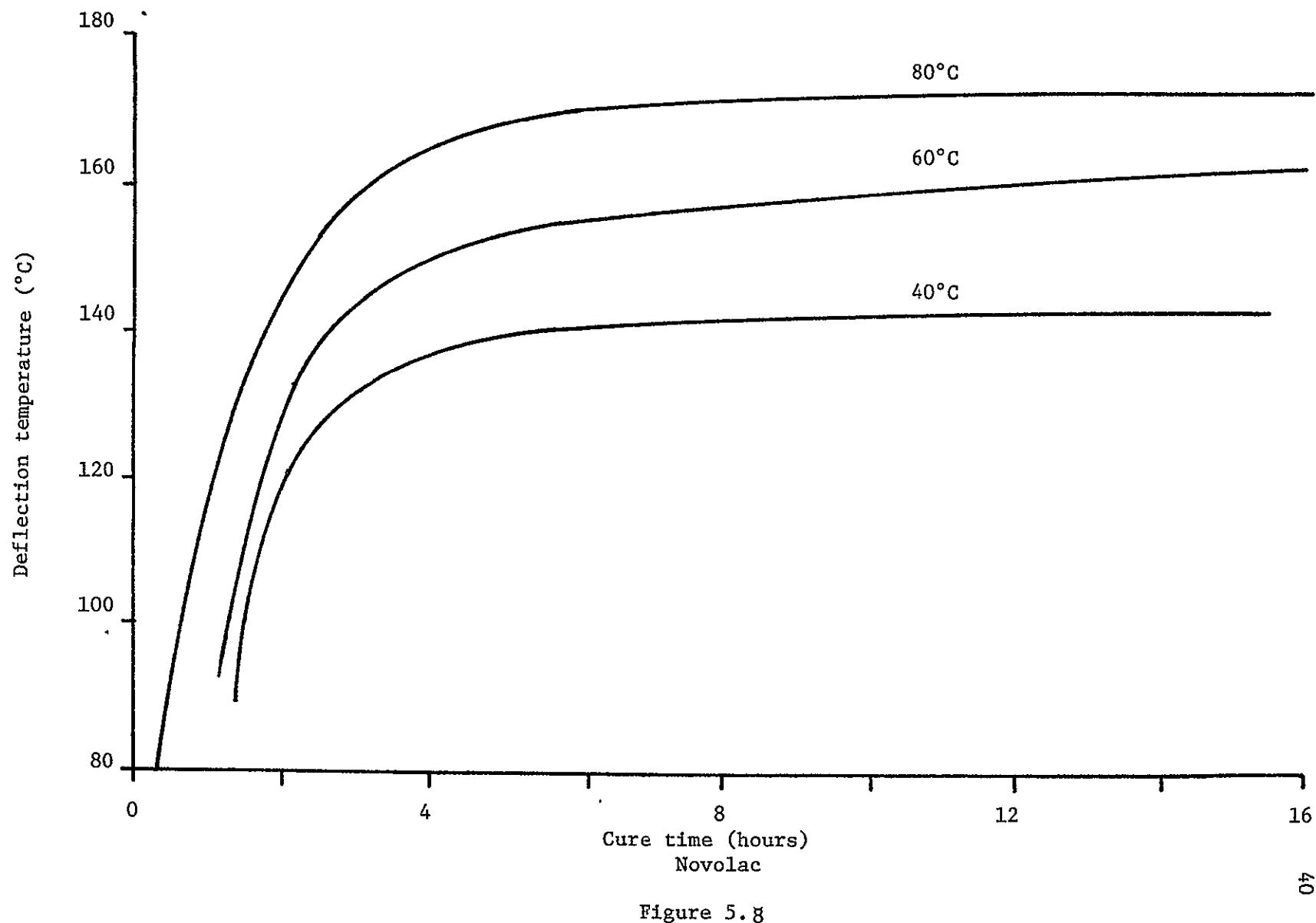
Figure 5.6

Figure 5.7

Figure 5.8

Thermal conductivity is the ability of a material to conduct heat. In general most unfilled epoxies have a thermal conductivity of approximately 500×10^{-6} cal/sec-sq cm-°C-cm. Fillers tend to increase the thermal conductivity; the extent of increase depends on the loading volume of the filler (Fig. 5.9).[9] Metallic fillers tend to increase the thermal conductivity to a greater extent than do the non-matallic fillers. However, the particle size and shape of the filler will primarily determine the thermal conductivity since they regulate the amount of filler used in a resin. In general, the thermal conductivity of an adhesive depends on the amount of filler used rather than on the type, hence high concentrations of fillers tend to increase the thermal conductivity.

From the foregoing discussion, it can be deduced that bisphenol based resins generally prove to be the most stable thermally without regards to the type of filler or catalyst used with them. Addition of just the right catalyst and filler can, however, improve thermal stability. The desired properties of the cured adhesives must now be considered. Catalysts and fillers should be chosen such that the cured adhesives yield the correct results for a particular application. For example, metallic fillers would not be used successfully when a thermally insulative adhesive is desired; or if the adhesive is to be used at predominately high temperatures, polyimides would be the best choice. Other factors such as heat distortion and thermal expansion must also be considered, particularly in cases where stress or strain is to be imposed on the adhesive.

70

60

50

Thermal conductivity x 10^4

40

30

20

10

0

42

Aluminum

Silica

Calcium Carbonate

0   20   40

Effect of Fillers on Thermal Conductivity
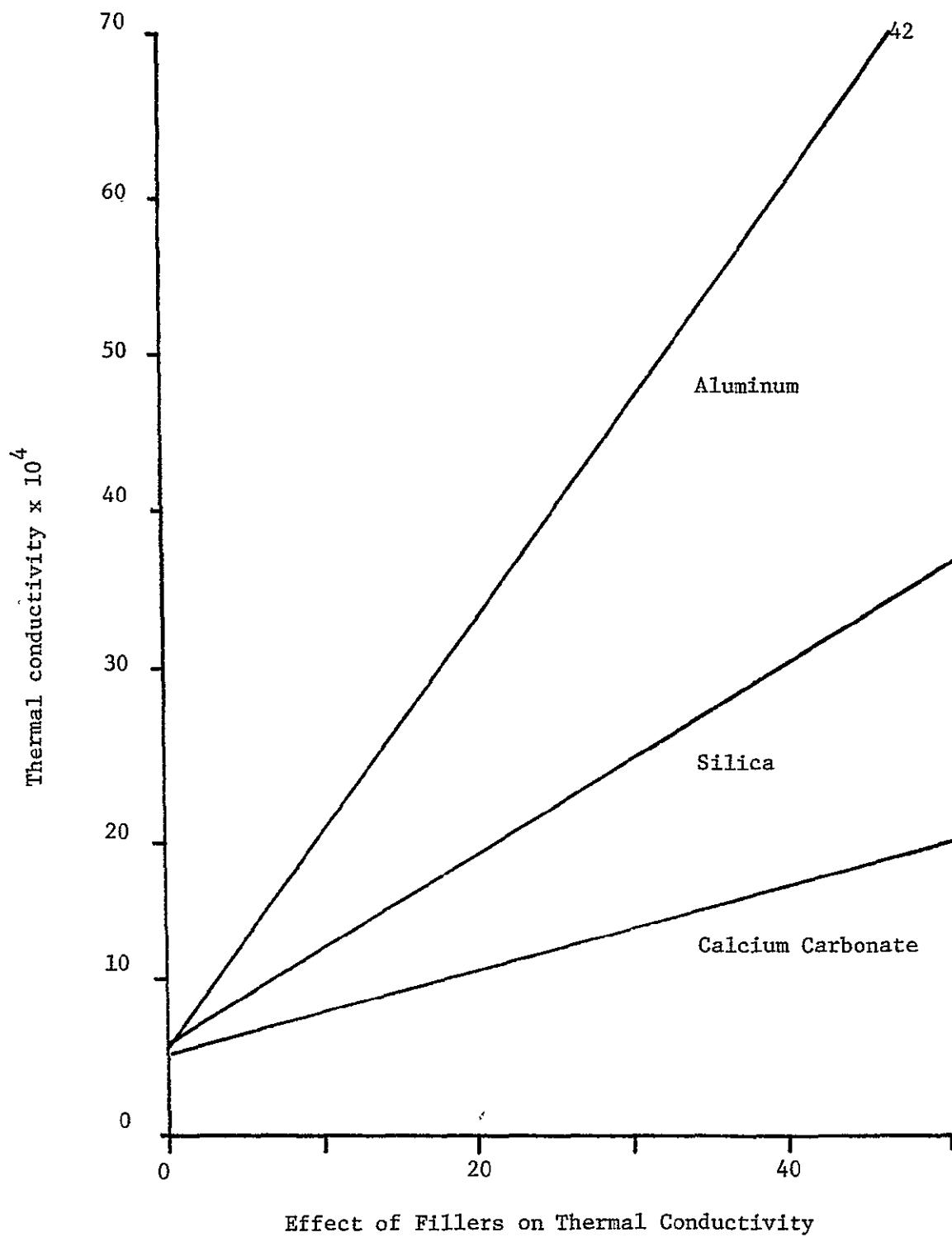
Figure 5.9

Footnotes

[1]Henry Lee and Kris Neville, ed., <u>Handbook of Epoxy Resins</u>
(New York, McGraw-Hill Book Company, 1967), p. 4.9.

[2]Ibid., p. 6.21.

[3]Charles A. Harper, ed., <u>Handbook of Plastics and Elastomers</u>
(New York, McGraw-Hill Book Company, 1975), p. 4.28.

[4]Lee, et al., p. 17.16.

[5]Charles A. Harper, <u>Electronic Packaging with Resins</u> (New York,
McGraw-Hill Book Company, 1961), p. 220.

[6]Lee, et al., p. 17.19.

[7]Ibid., p.6.26.

[8]Ibid., p. 8.7.

[9]Ibid., p. 14.22.

Chapter VI

Mechanical Properties of Adhesives

Of the mechanical properties to be discussed in this chapter, adhesion is perhaps the most important, followed closely by hardness and compressibility. Tensile and shear strength, which are predominately, used to characterize adhesion, will also be discussed. These properties will also be examined in Chapter IX in reference to some particular adhesives used in microelectronics.

Adhesion is the ability of two surfaces to hold together by some interfacial force. Epoxies are excellent adhesives because of their chemical structure. The hydroxyl and ether groups of adhesives allow maximum surface contact between the two surfaces by reducing shrinkage. Shrinkage is the dimensional change in a material that occurs over a certain period of time usually during curing. Figure 6.1 shows the realtionship between curing temperature and shrinkage.[1] Shrinkage may be caused by reaction and rearrangement of molecules into a more compact configuration or by cooling the material from high temperatures. Fillers, by bulk displacement, can also reduce shrinkage, thereby increasing adhesive strength. Catalysts may also affect shrinkage as shown in Figures 6.2 and 6.3.[2]

Tensile and shear strength also characterize adhesive properties. Tensile strength is the amount of pull required to break a sample of material. In general, tensile strength is increased with the use of fibrous fillers such as milled glass, but is decreased with the use

Per cent linear shrinkage

Figure 6.1

The y-axis is labeled "Curing temperature (°C)" with values 0, 120, 140, 160, 180, 200, 220, 240, 260. The x-axis is labeled "Per cent linear shrinkage" with values 0, 0.8, 1.6, 2.4, 3.2, 4.0.
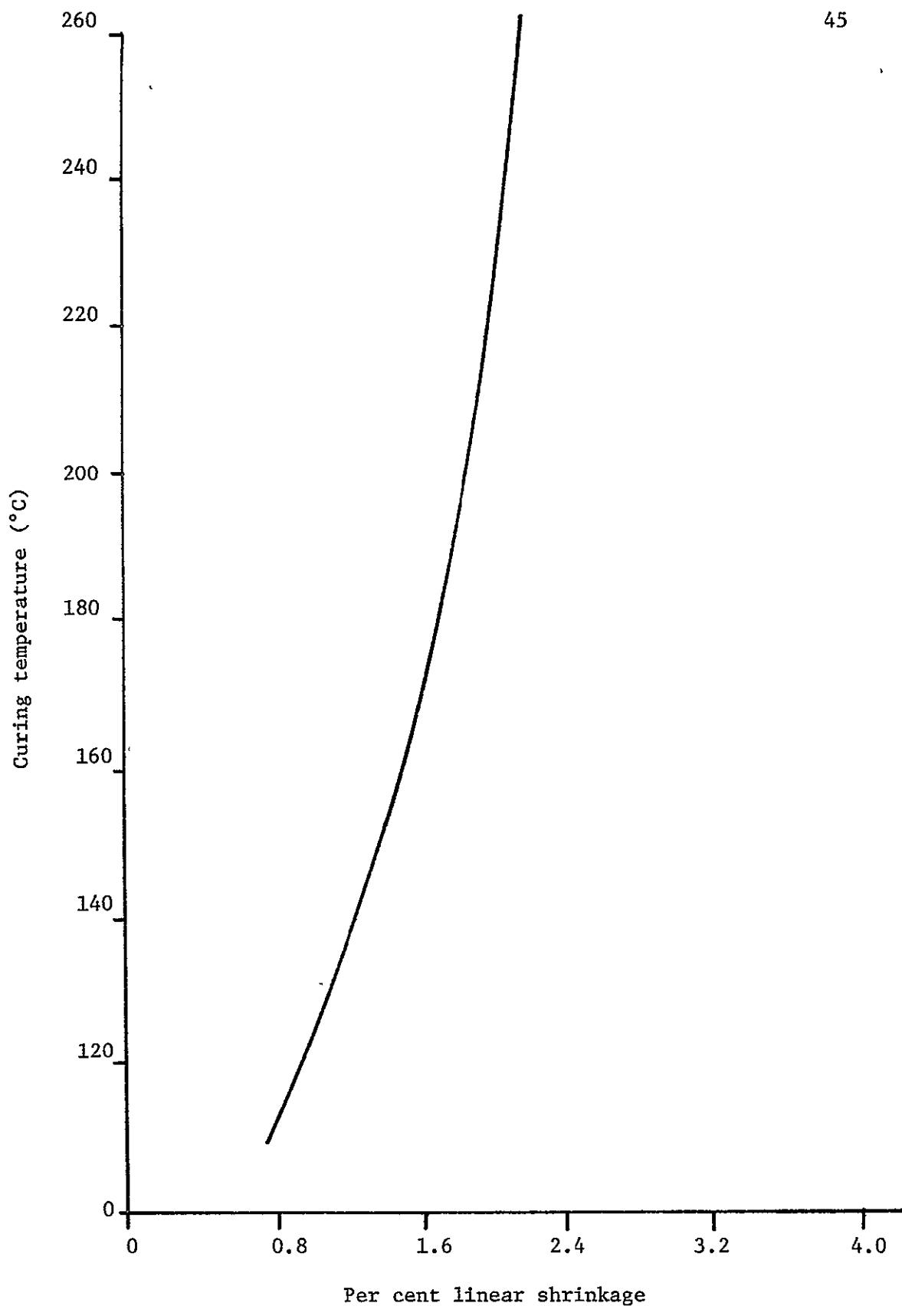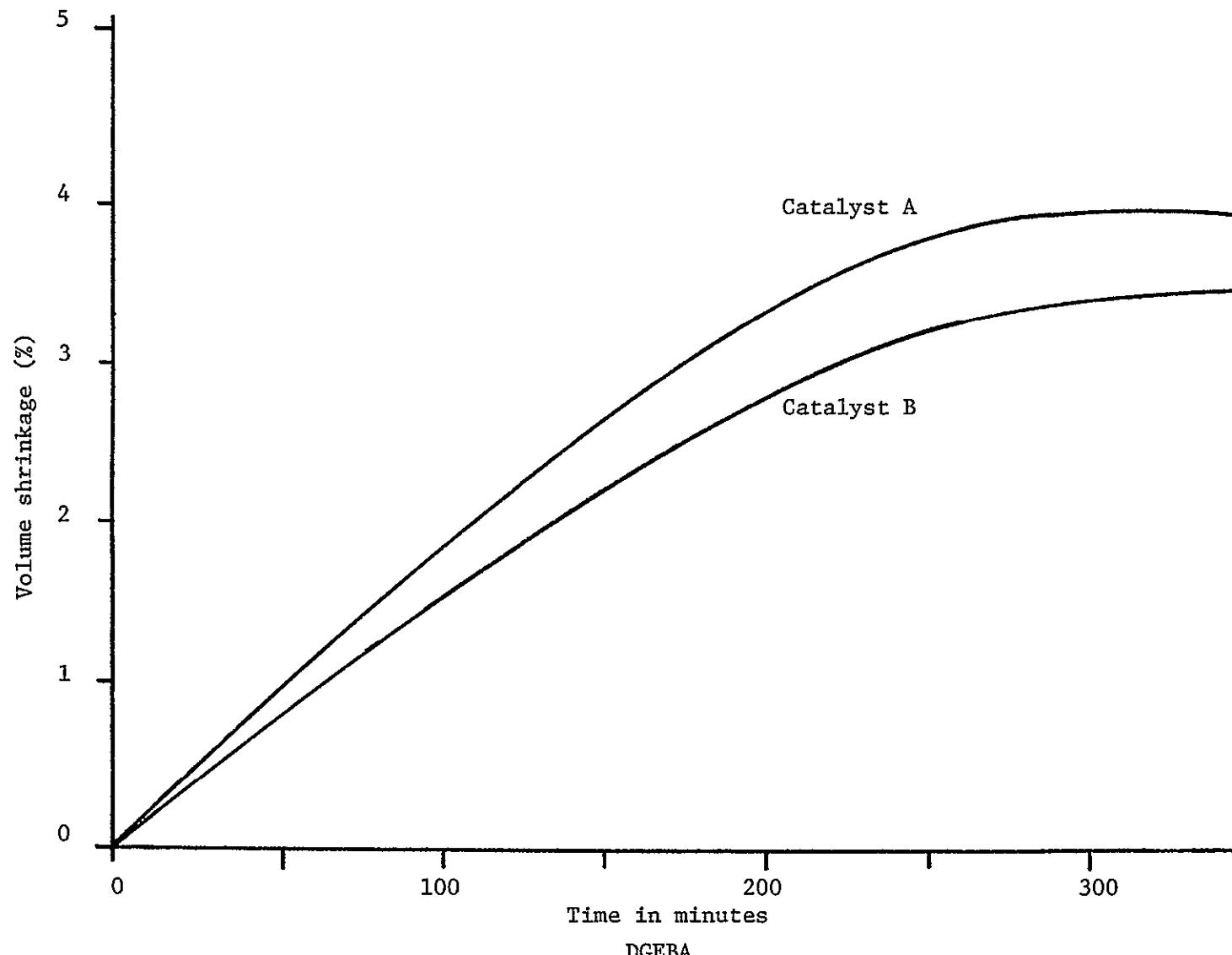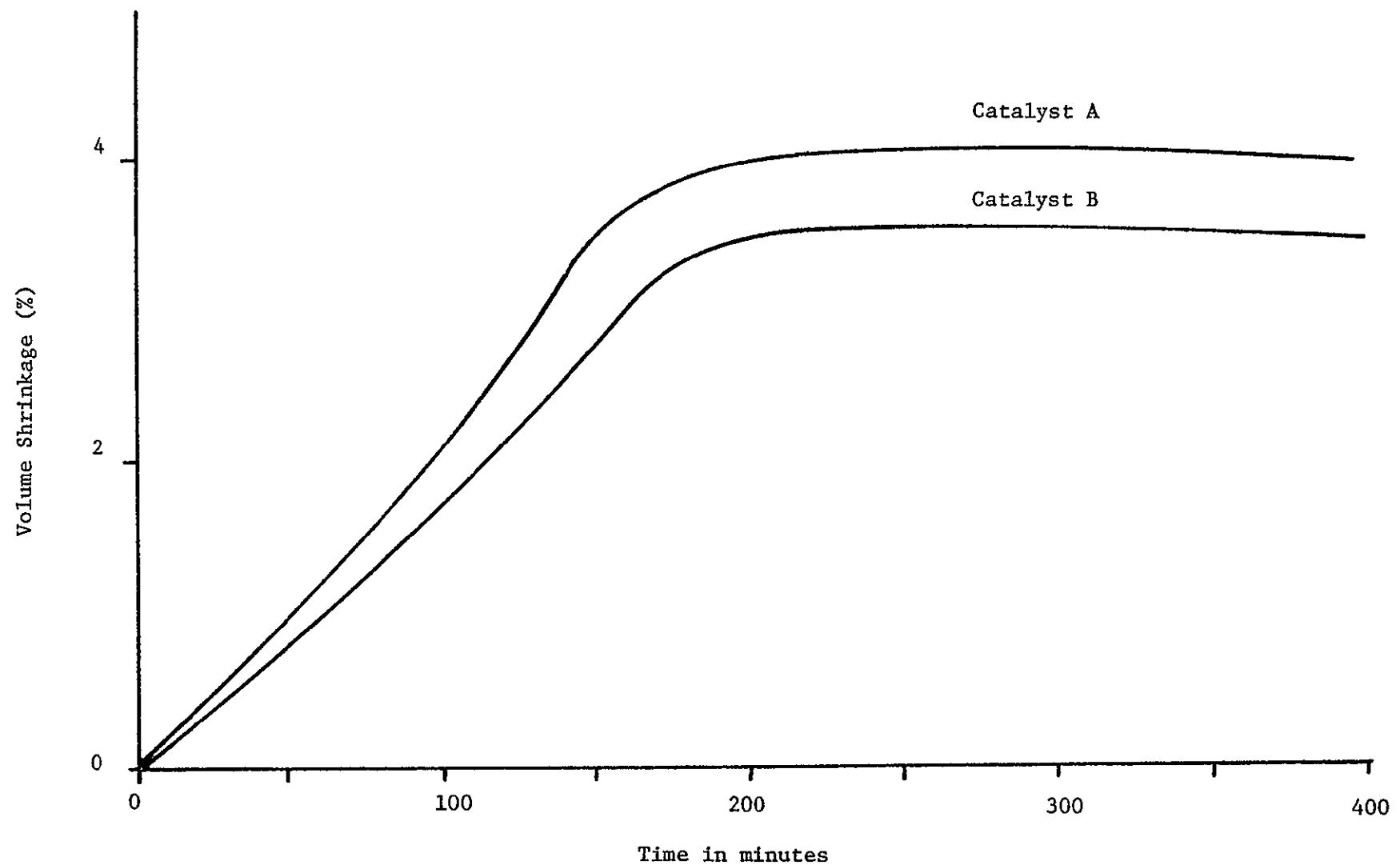
Figure 6.2

Figure 6.3

of bulk fillers. However, fibrous fillers in low concentrations may also reduce tensile strength. Improvements in tensile strength are generally noticed when concentrations of fillers are greater than ten per cent by volume. Tensile strength decreases are more evident in amine-cured resins than in anhydride-cured resins. Anhydride-cured resins sometimes show marginal increases in tensile strength. Tensile strength is also often increased by using blends of resins rather than just the pure resin by itself (Fig 6.4).[3] Tensile strength has been shown to correlate with some accuracy to deflection temperature. The higher-temperature systems have been found to have low tensile strengths. This is attributed to the flaw sensitivity of the more tightly cross-linked glycicyl ether systems of the epoxies.

Shear strength is the amount of torsion stress that a material can withstand. Shear strength increases slowly during the formation of branched and linear molecules, then increases very rapidly near the end of the reaction or formation of cured networks. Figure 6.5 shows the shear strength of a typical adhesive for a particular curing time and temperature.[4] Lap shear strength is dependent on the amount of curing agent as indicated in Table 6.1.[5]

Hardness is the ability of a material to withstand localized indentation. Rockwell hardness is perhaps the most common measure of hardness. Durometers of the Shore type are used to measure indentations in softer adhesives. Another tester for measuring hardness in the Rockwell range is the Barcol hardness tester. Fillers, in general, increase hardness, the amount varying from filler to filler. Unfilled epoxies, for example, have a Rockwell hardness of M80 to M110 while silica filled epoxies have a hardness of M85 to M120.[6] Closely
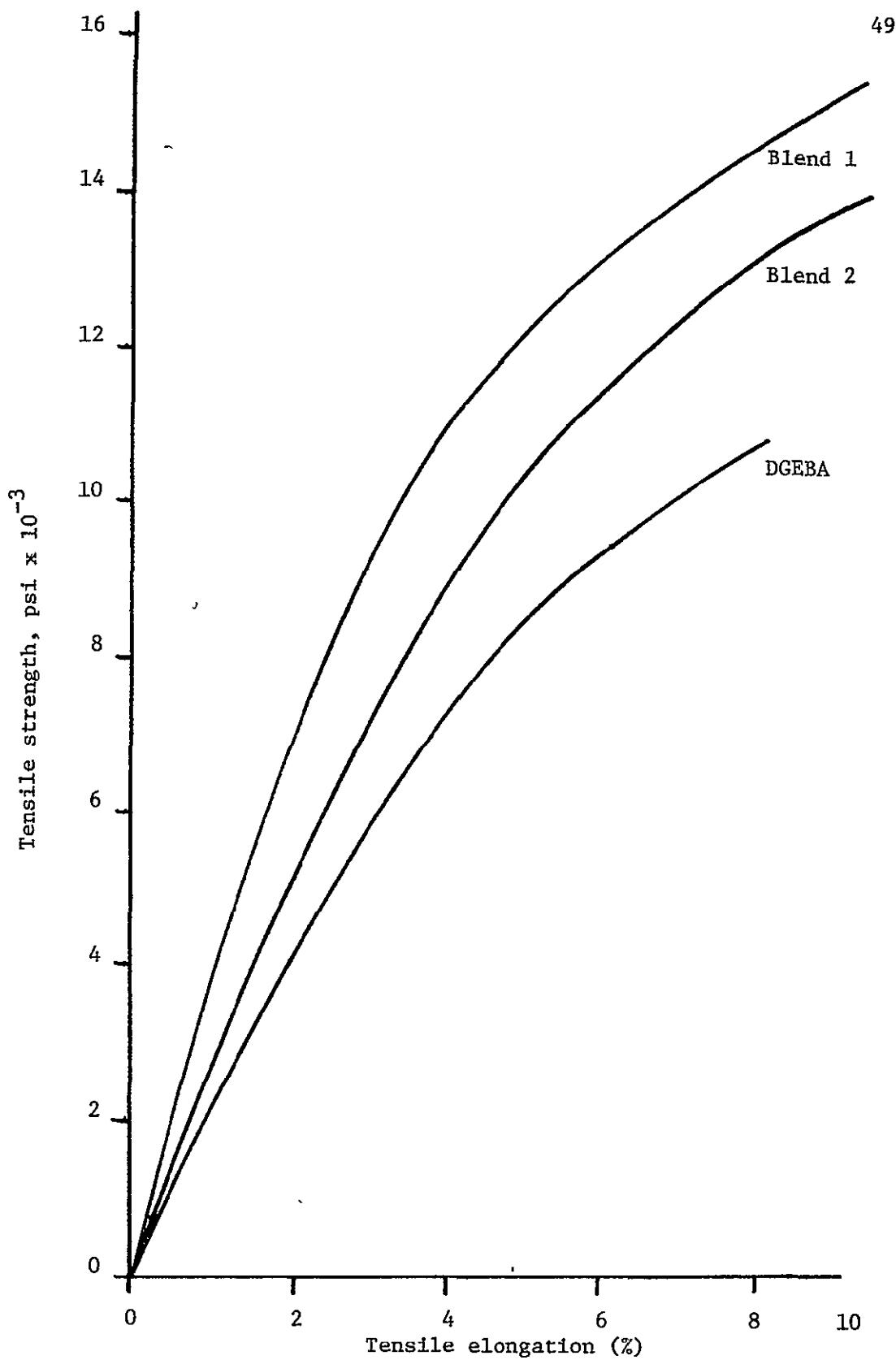
Figure 6.4
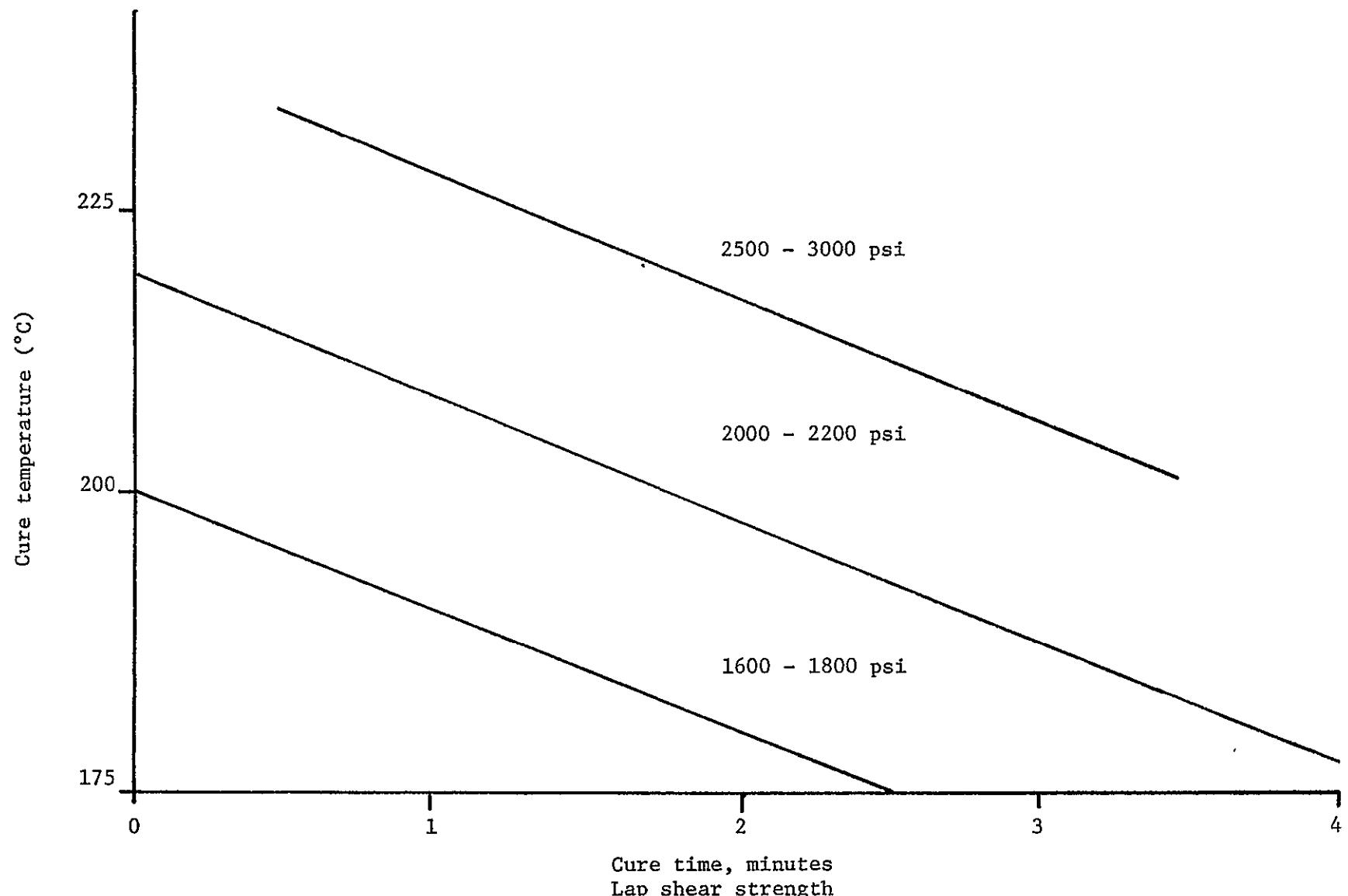
Figure 6.5

Table 6.1

Curing Agent Concentration Parts Per Hundred Resin	Adhesive Strength Lap Shear--p.s.i.
10	600- 700
15	2000-2100
20	2000-2100
25	2100-2200
35	230h-2500

Cure cycle of 60 minutes at 150°C.

associated with hardness is the compressive strength of a material to withsta-d a crushing force. Compressive yield is the point at which deformation continues without further loading. Fillers generally increase compressive yield strength because of their stiffening effect, the amount of increase depending on the type and amount of filler used (Figs. 6.6 and 6.7).[7] Typical compressive strengths for epoxy resins are in the area of 15,000 to 30,000 lb/in^2.[8]

Most mechanical properties are seen to be primarily dependent on the amount and type of filler used. Most properties can be improved with filler concentrations greater than ten per cent by volume. However, the compressive strength decreases with decreasing hardness of filler. Silica and alumina provide for better compressive strength than do the softer metals such as silver and gold. The effect of fillers on the mechanical properties of particular adhesives used in microelectronics will be examined in Chapter IX.

Compressive strength of DGEBA

Figure 6.6

Effect of filler concentration on compressive yield
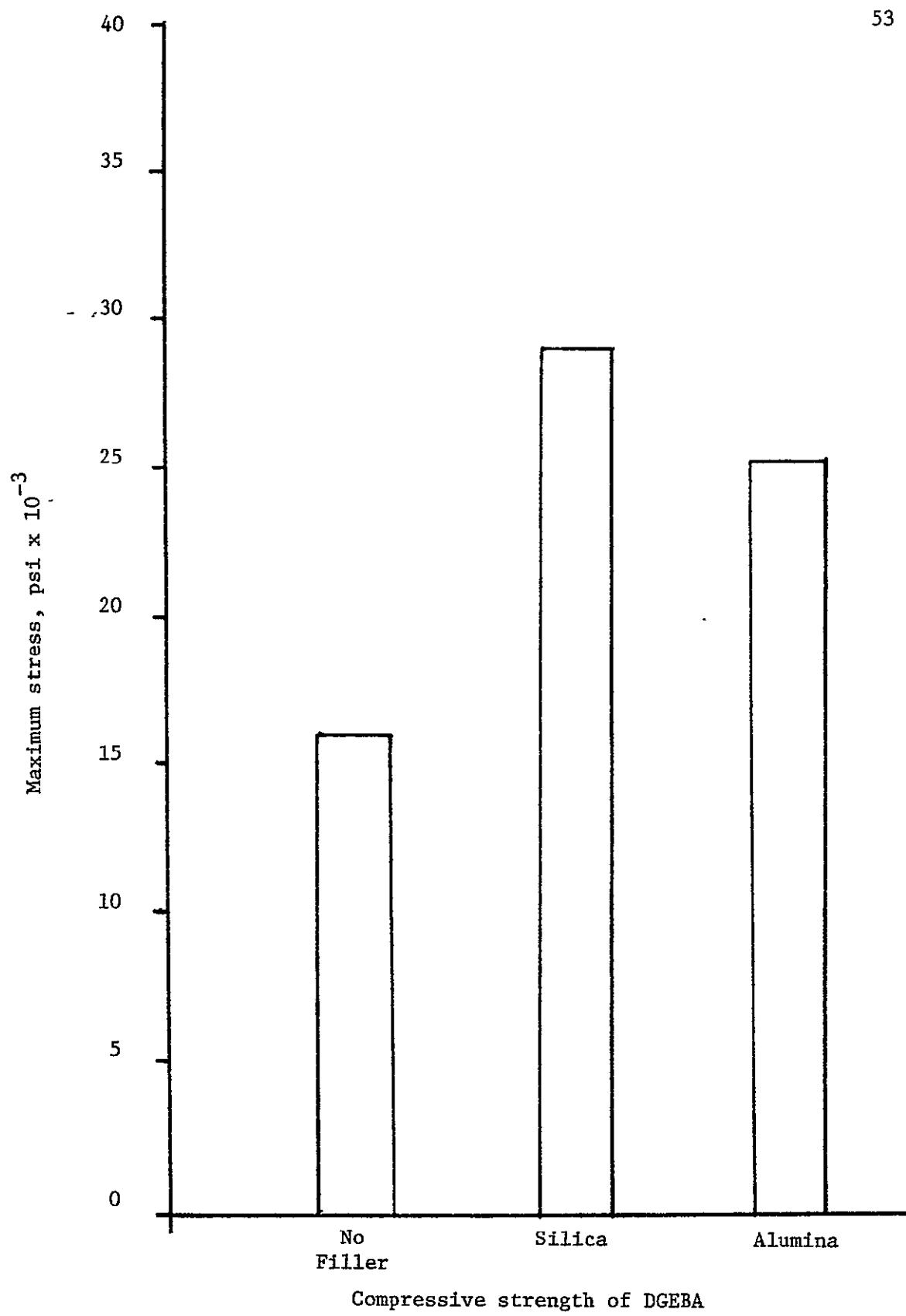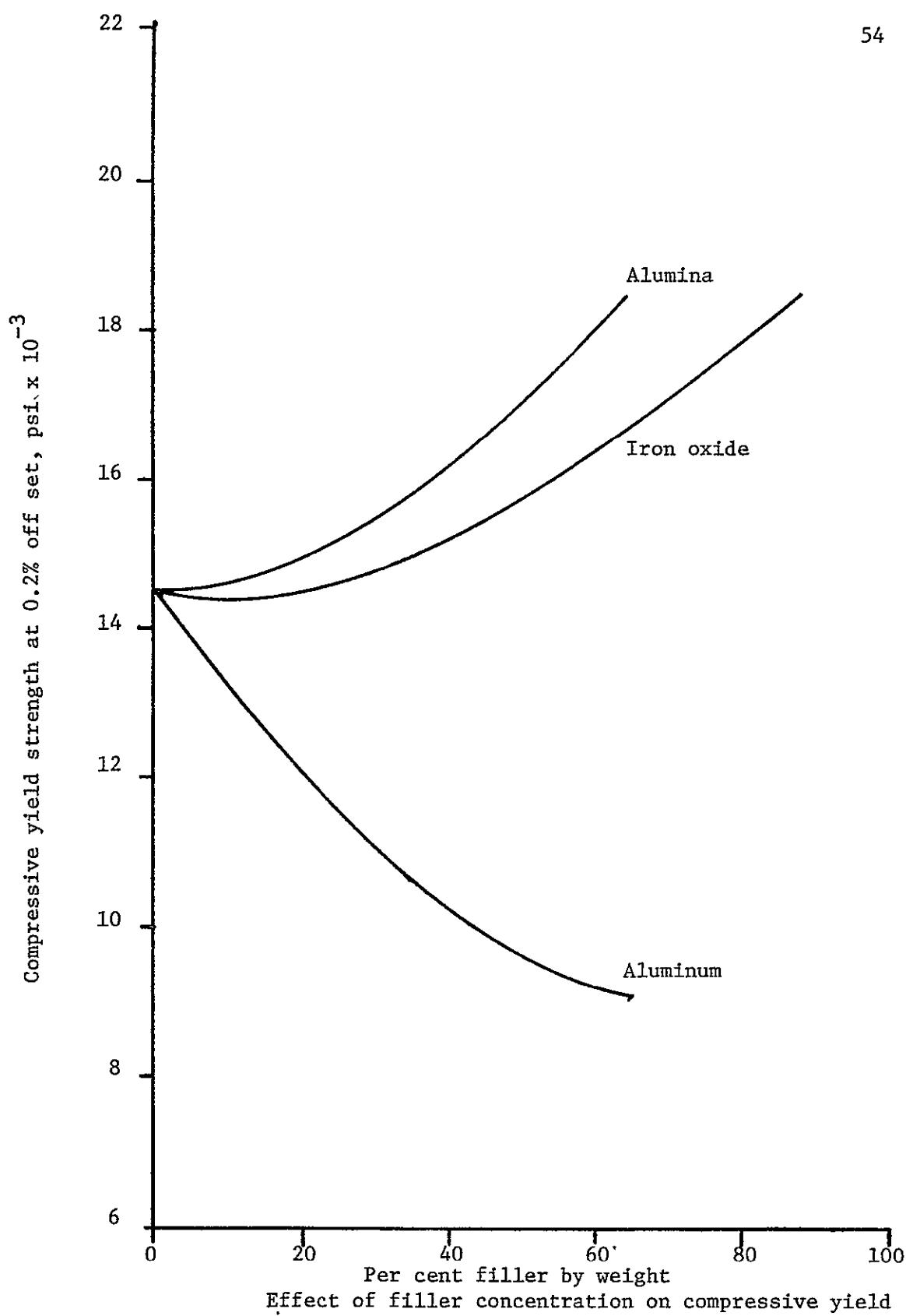
Figure 6.7

Footnotes

[1]Henry Lee and Kris Neville, ed., <u>Handbook of Epoxy Resins</u>
(New York, McGraw-Hill Book Company, 1967), p. 12.18.

[2]Ibid., p. 17.12.

[3]Ibid., p. 8.15.

[4]Robert F. Gould, ed., <u>Epoxy Resins</u>, (Washington, D.C.,
American Chemical Society, 1970), p. 170.

[5]Ibid.

[6]Charles A. Harper, ed., <u>Handbook of Plastics and Elastomers</u>
(New York; McGraw-Hill Book Company, 1975), p. 3.56.

[7]Lee, et al., p. 14.19-

[8]Harper, p. 3.5.

Chapter VII

Electrical Properties of Adhesives

Until recent years industries were concerned primarily with the
insulating properties of adhesives used in electronics. As electronic
circuits became progressively smaller, modes of bonding such as
soldering became increasingly more difficult to use. Conductive ad-
hesives are now beginning to replace soldering and other techniques of
bonding in microelectronics. However this chapter will deal with the
electrical properties of adhesives in general.

Failure of adhesives is usually characterized by an excessive
flow of current or an arc and by partial destruction of the adhesive.
All adhesives fail at some level of applied voltage for a given set of
operating conditions. Dielectric strength, measured in volts/mil, is
the voltage that an adhesive can withstand before breakdown occurs--
the higher the value of dielectric strength, the better the insulating
properties. In general, dielectric strength increases as the thickness
of the sample decreases. Dielectric strength is also influenced by
temperature, humidity, voids or foreign materials in the adhesive,
frequency, and geometry. Figure 7.1 shows the dielectric strength
of adhesives versus temperature of polyimides.[1] Fillers change the
dielectric strength only slightly as shown in Table 7.1 and Figure 7.2.[2]

The dielectric constant of an adhesive is the ratio of the capaci-
tance formed by two parallel plates with the adhesive between them to the
capacitance of the two plates with air between them. For a typical

Polyimides
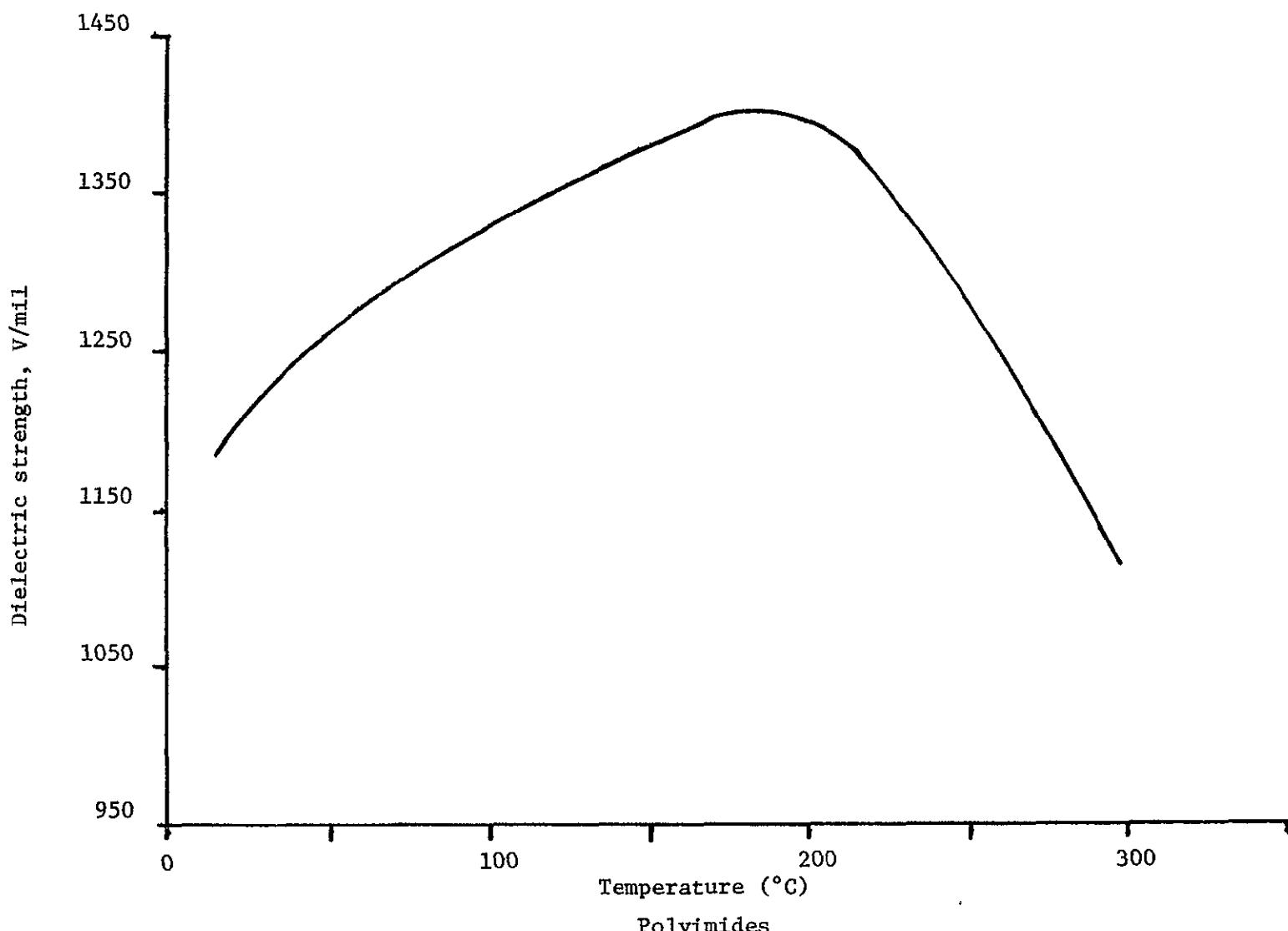
Figure 7.1

Table 7.1

Dielectric Strength of DEGBA as a Function of Filler, Loaded to
Provide an Initial Viscosity of 4,000 to 5,000 Centipoises at
Working Temperature and Cured with Stoichiometric Amounts of
Listed Curing Agents.

Filler	DETA	MDA	HHPA	Anhydride
no filler	390	435	390	310
Silica	335	440	460	425
Alumina	410	400	350	360

(In Volts per Mil)

DGEBA
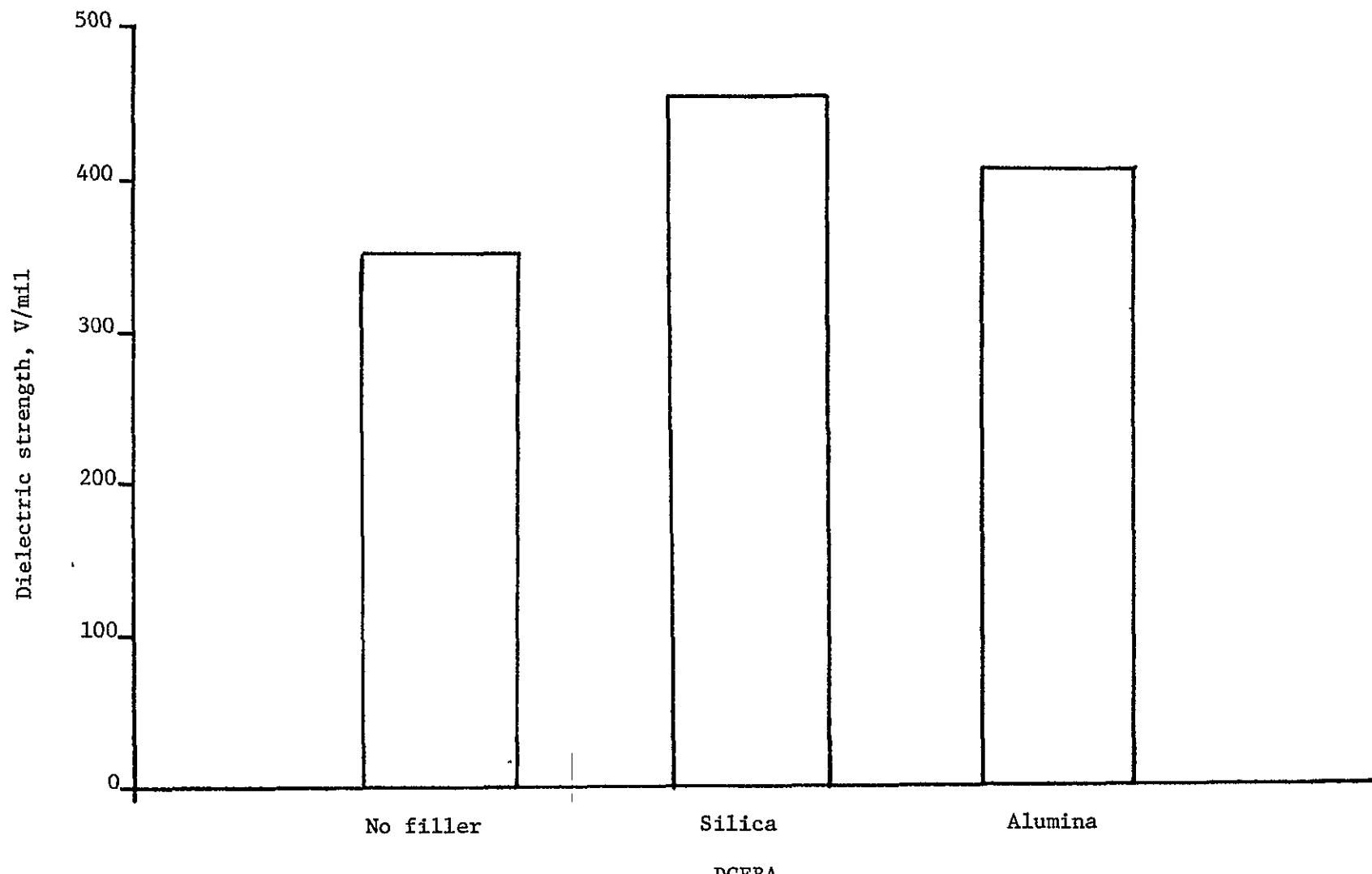
Figure 7.2

epoxy, the dielectric constant is approximately 3.8 as compared to that

of air which is normally taken as 1.[3] Low values of dielectric constant

are best for use in high-frequency or power applications to minimize

power loss. Higher values are better for use in capacitor applications.

For most adhesives, dielectric constants increase with temperature, es-

pecially above a critical temperature unique to each particular adhesive

(Fig. 7.3).[4] Fillers also generally increase the dielectric constant

with only slight differences seen between fillers (Fig. 7.4).[5] Water

content and humidity also tend to increase the dielectric constant of

adhesives (Figs. 7.5, 7.6, 7.7).[6] The dielectric constant also varies

with the type of curing agent used (Fig 7.8).[7] An increase in frequency,

however, will decrease the dielectric constant as is evident in Figure 7.9.[8]

The dissipation factor is the tangent of the loss angle. For

low values of the dissipation factor, it is almost equivalent to the

power factor and thus used interchangebly here. Power factor is the

ratio of power dissipated to the power input. For capacitor systems,

the power factor is a measure of the dielectric loss. The dissipation

factor, like the dielectric constant, is also a function of frequency

and temperature. Dissipation factors tend to increase with increasing

temperatures and decrease with increasing frequency (Figs. 7.10 and 7.11).[9]

Water and humidity have very little effect on the dissipation factor

as shown in Figure 7.12.[10] Fillers generally tend to increase the

dissipation factor. Oxide fillers, when added to silica filled epoxies,

are shown to lower the dissipation factor (Fig. 7.13).[11] Curing agents

also affect the deflection temperature as seen in Figure 7.14.[12]

Resistivity is the ability of a material to resist passage of

an electric current. Typical resistivity of an insulating adhesive is

Figure 7.3

DGEBA
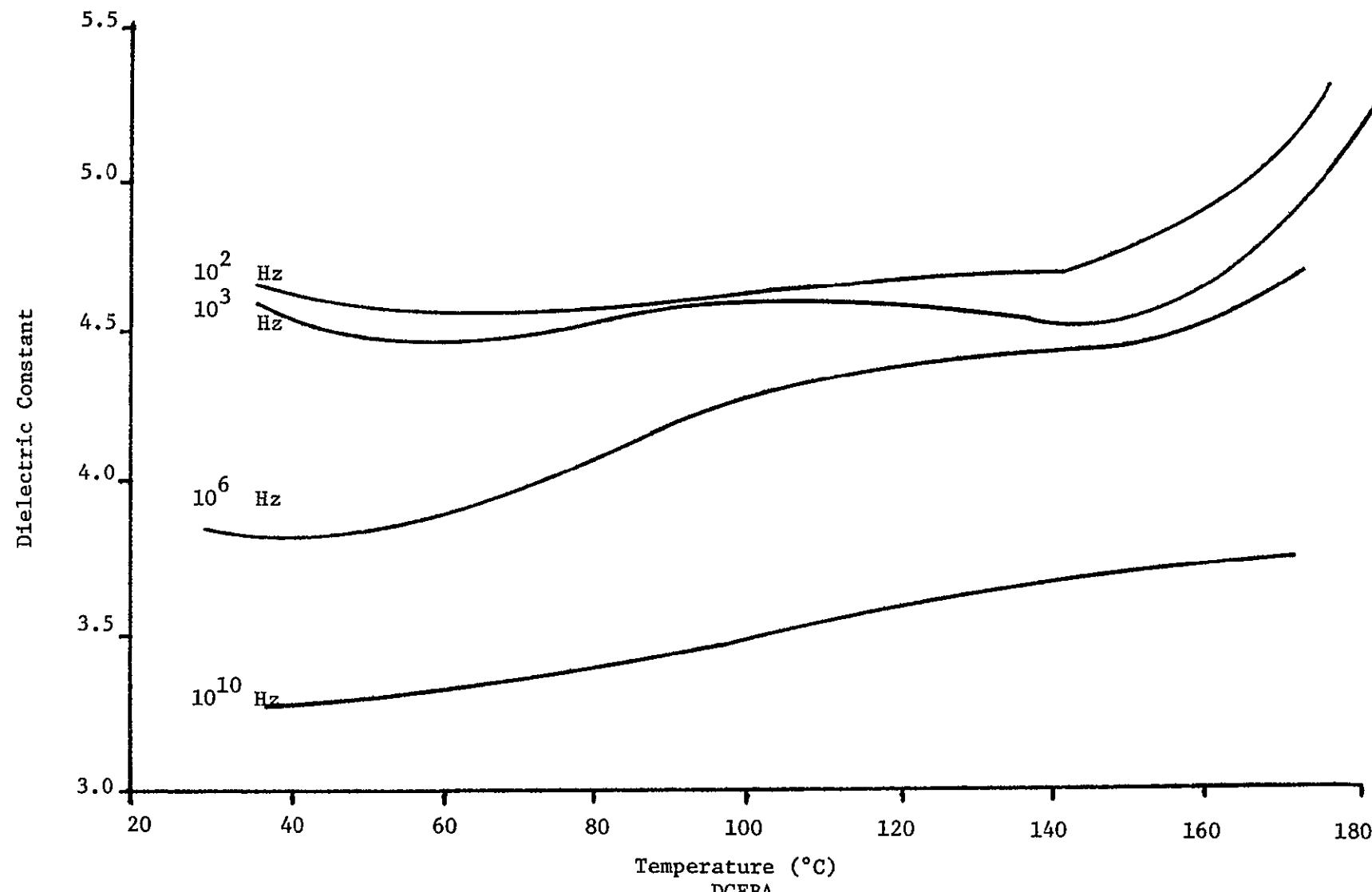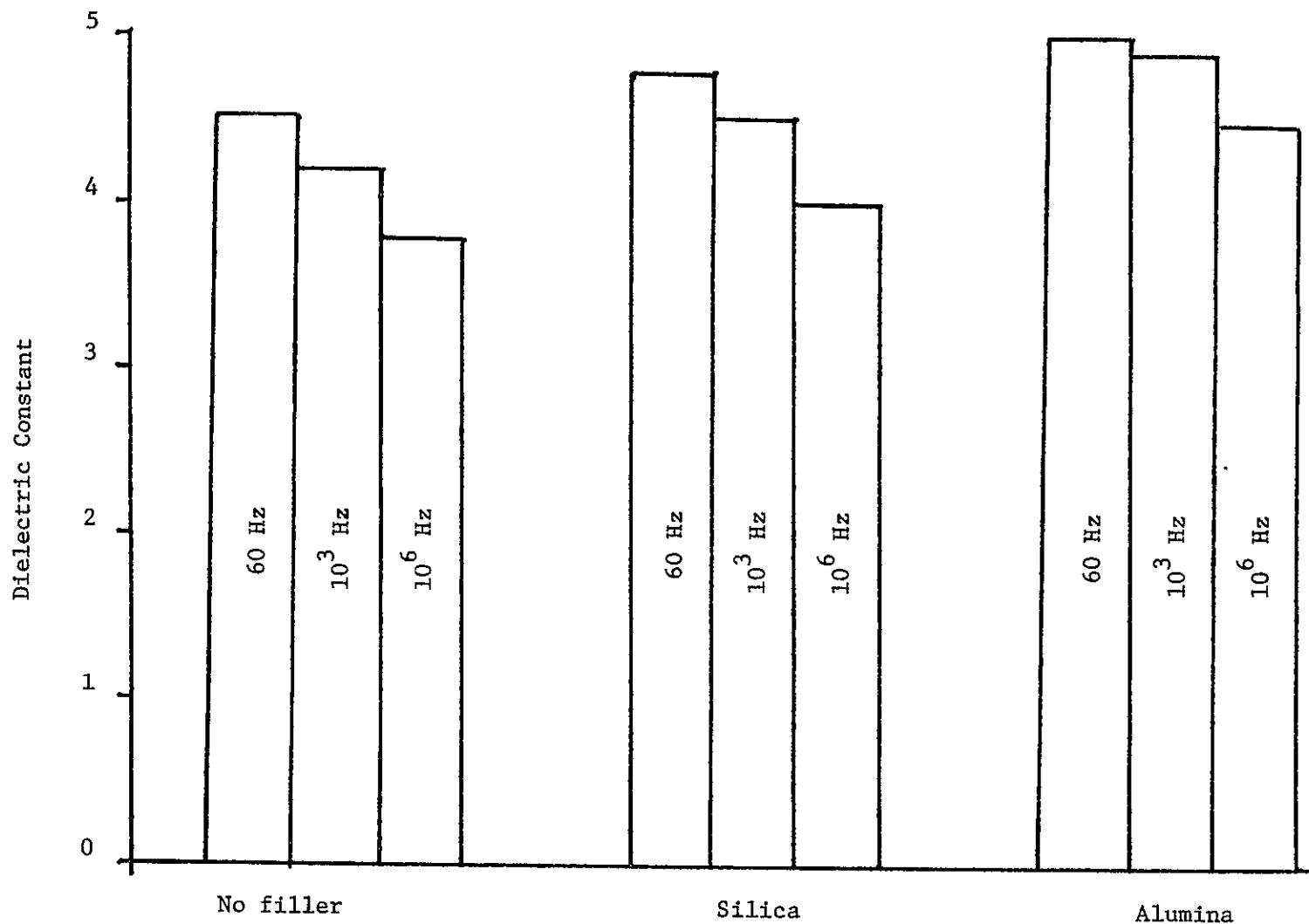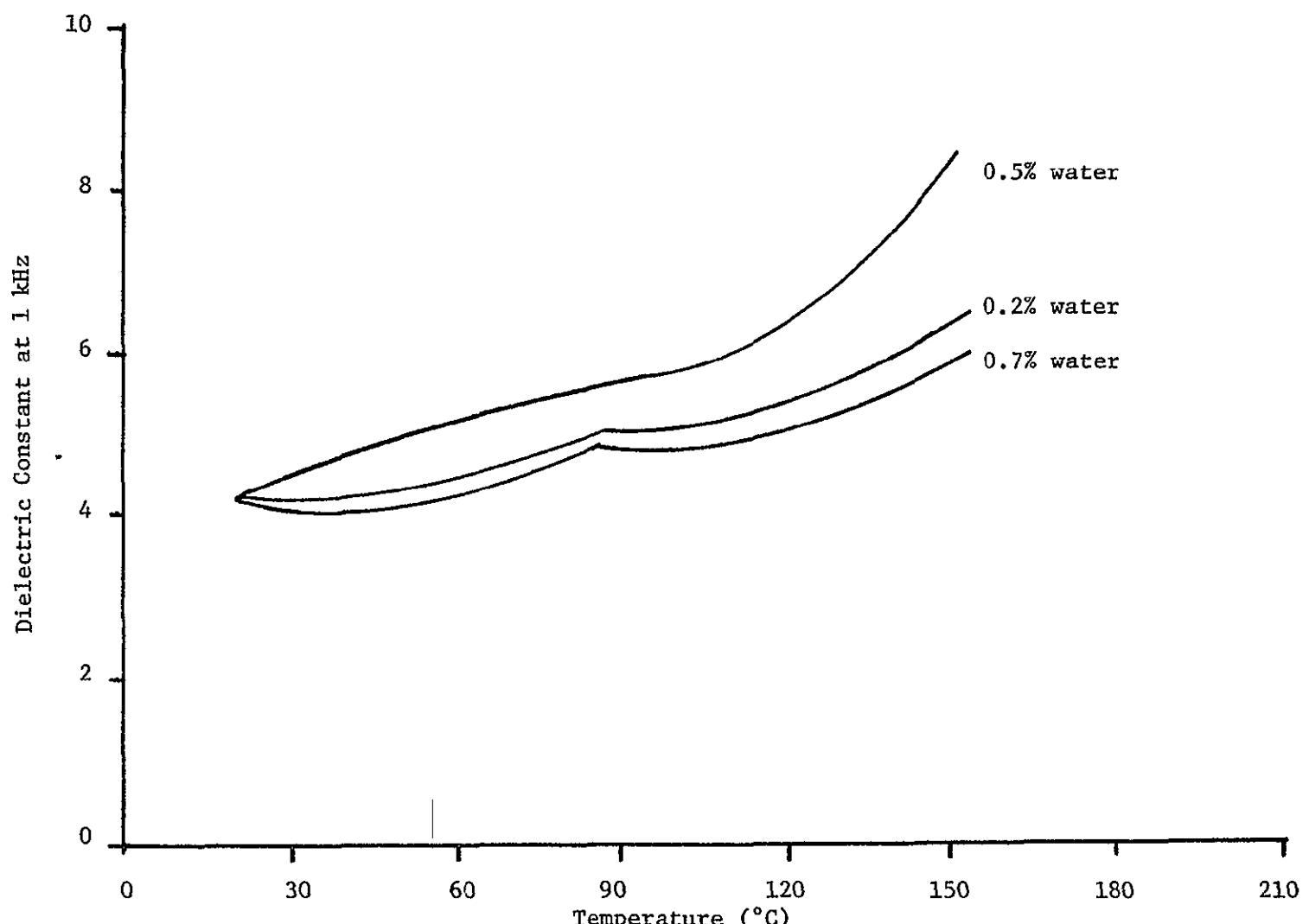
Figure 7.4

Figure 7.5
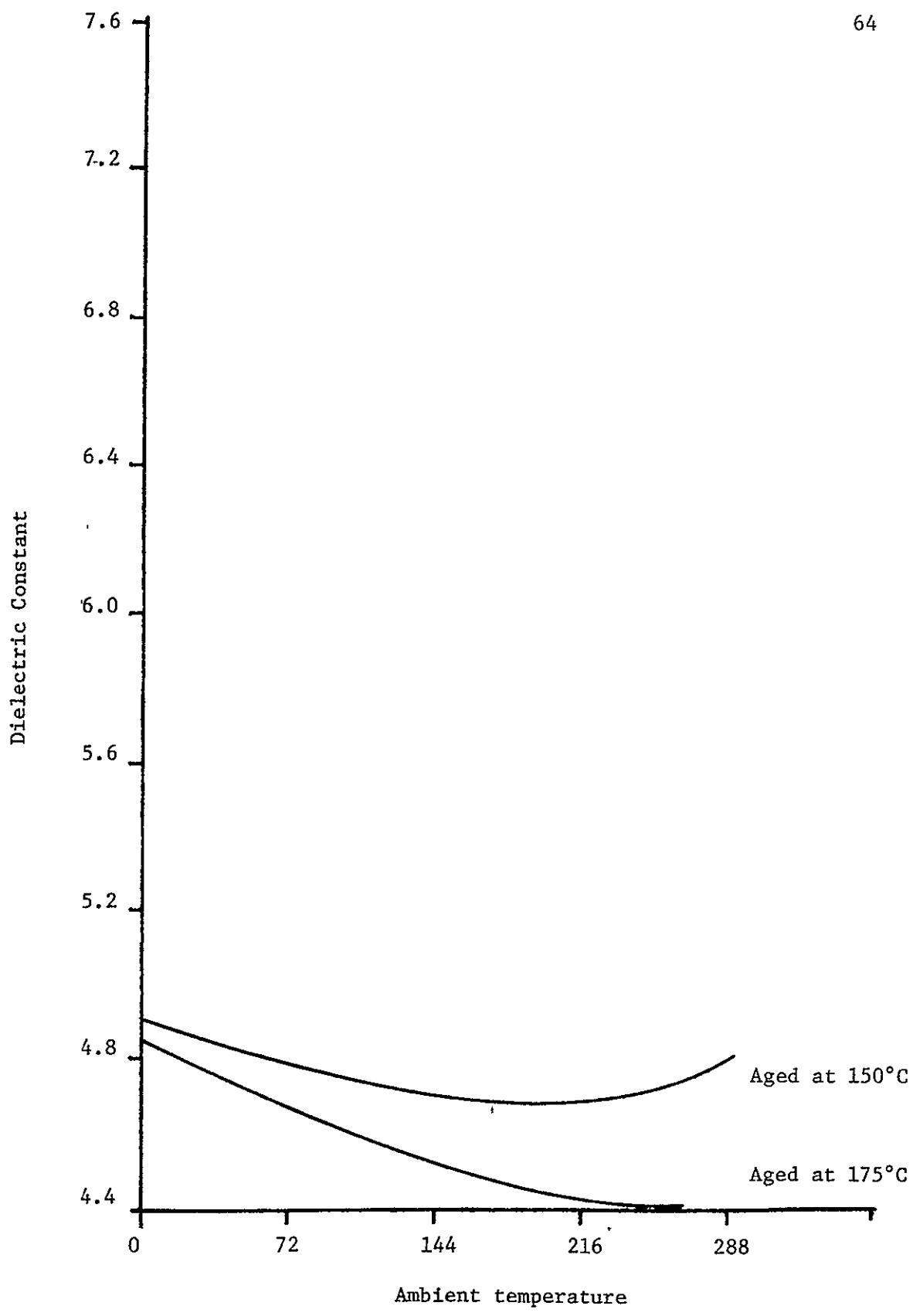
Figure 7.6
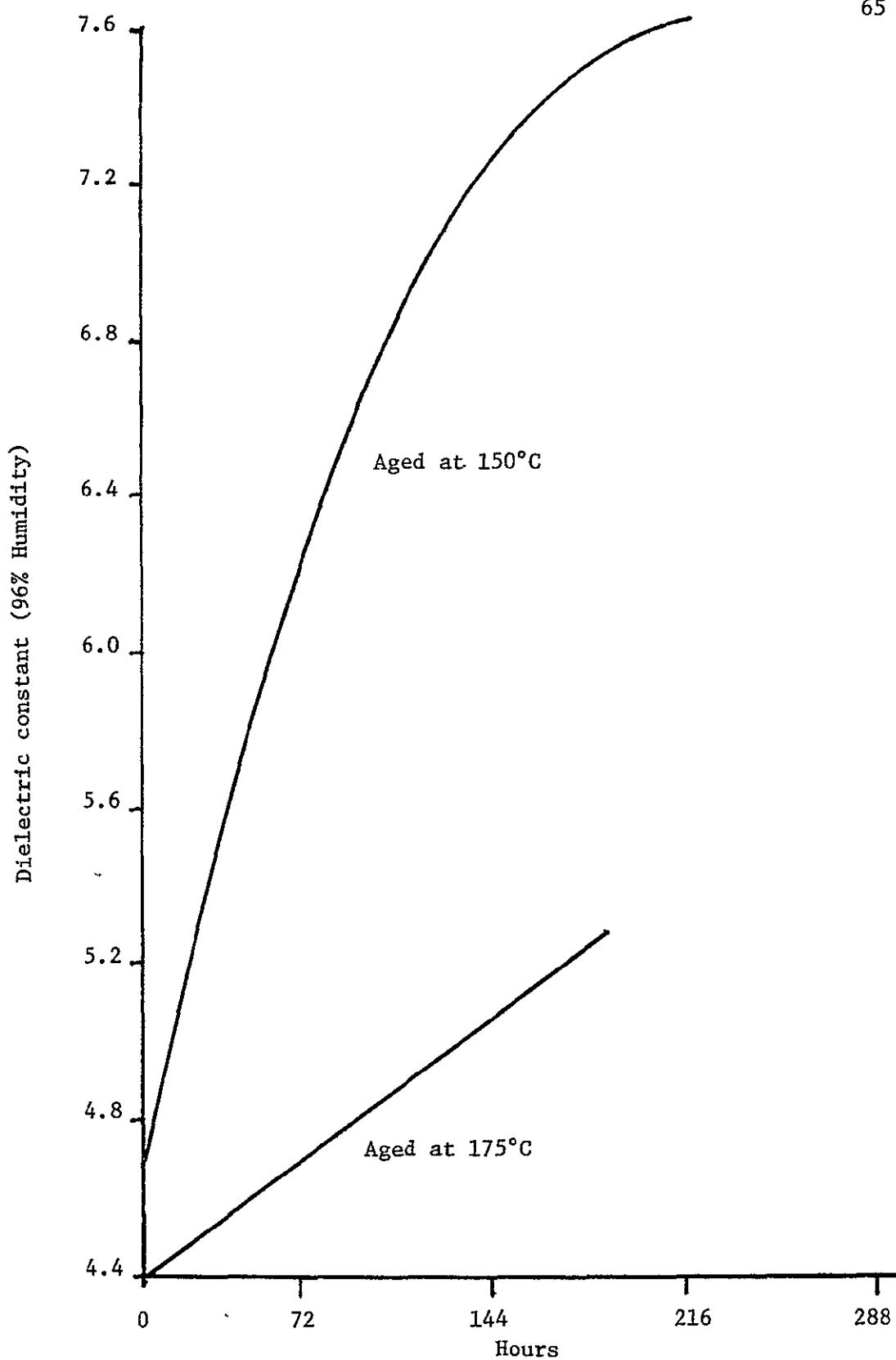
Figure 7.7
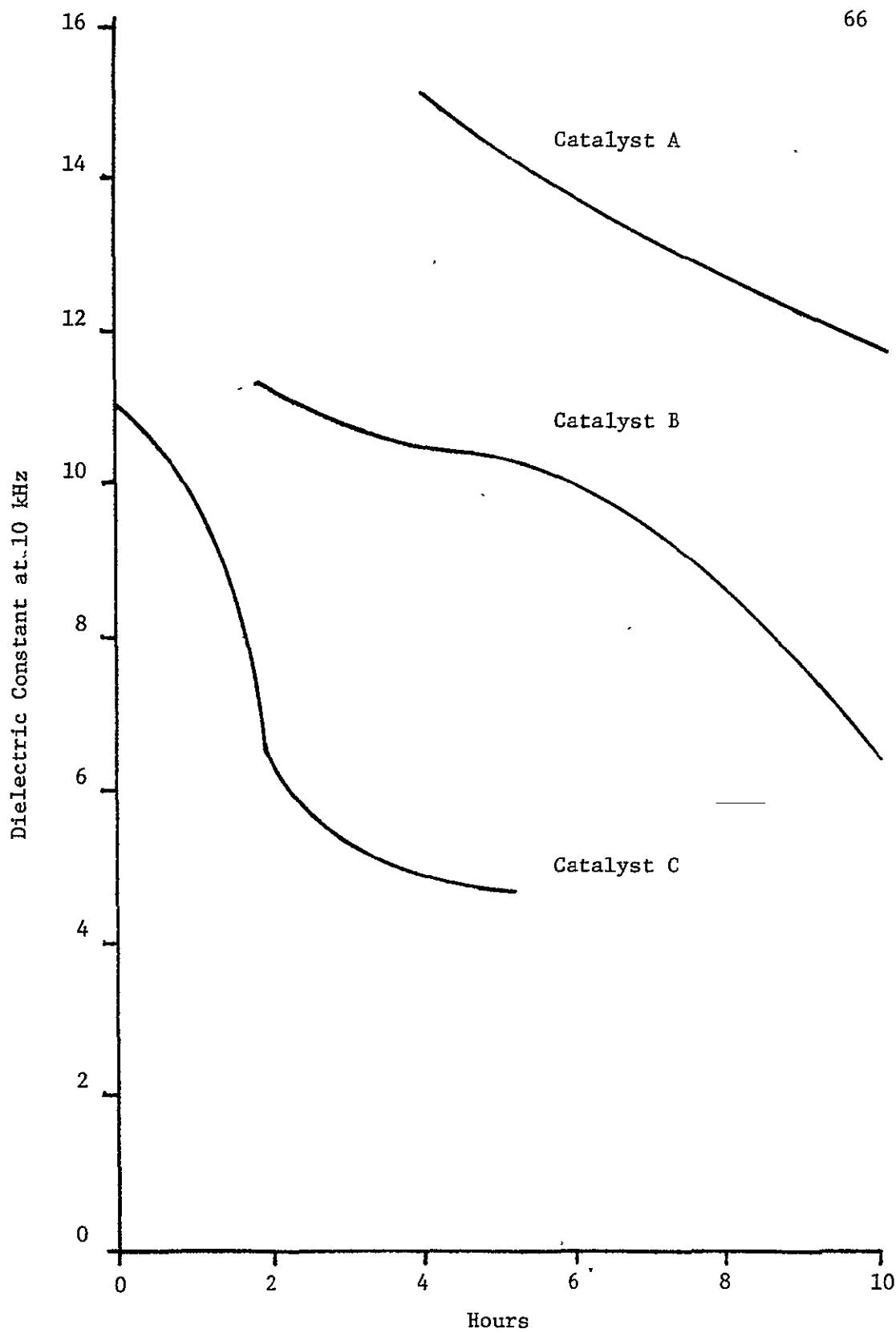
Figure 7.8
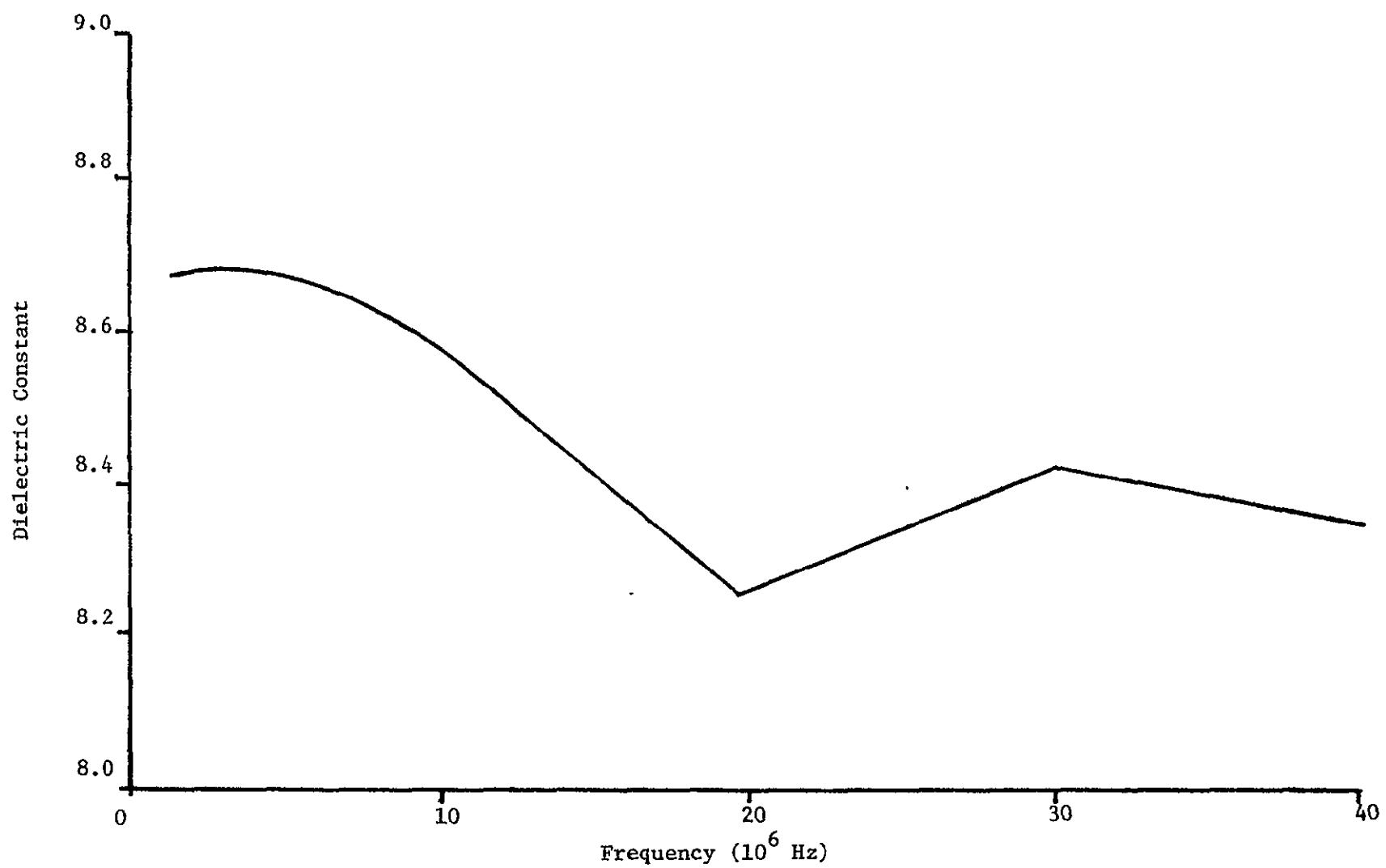
Figure 7.9

Figure 7.10

Figure 7.11

Effect of moisture on dissipation factor
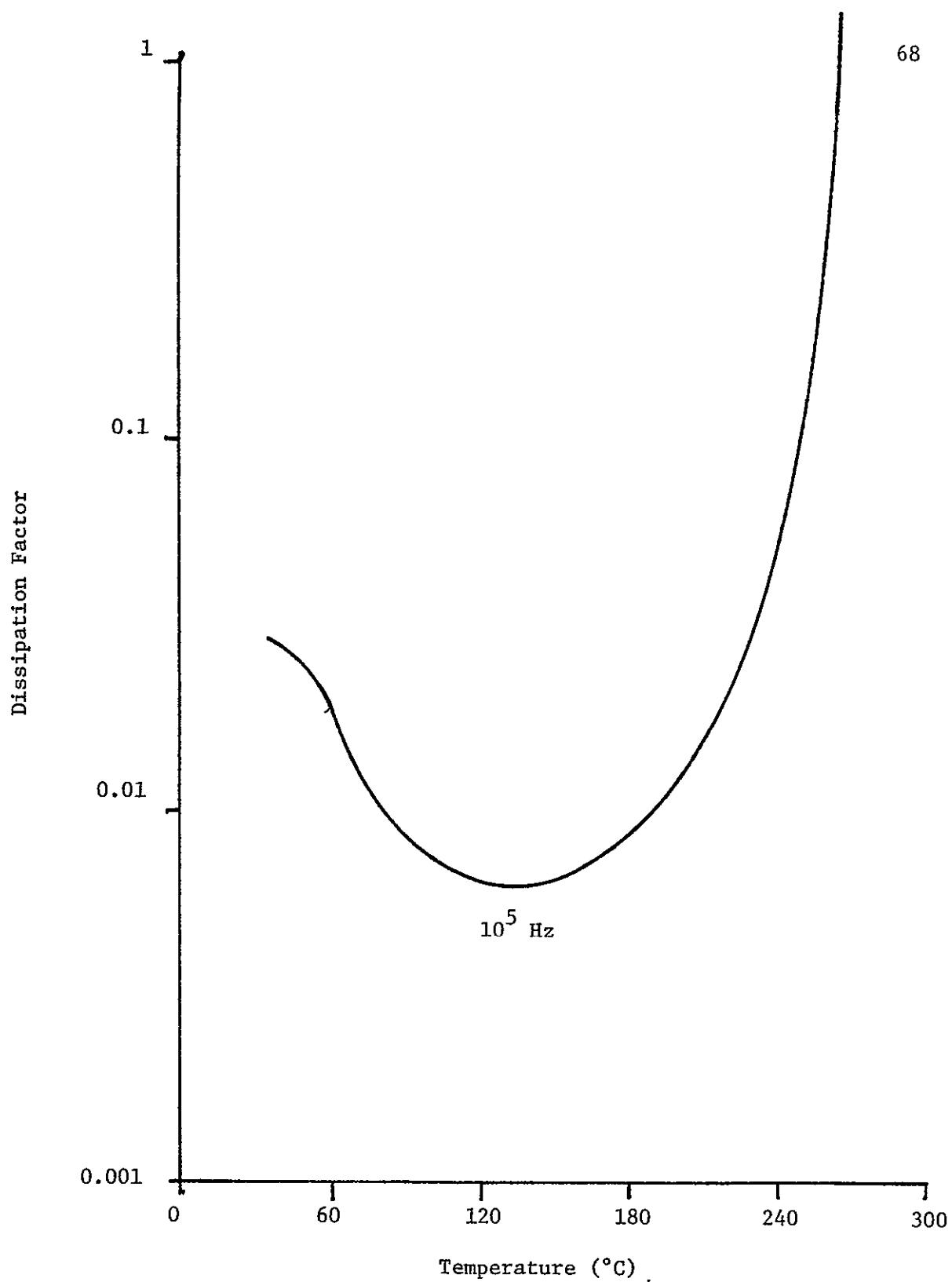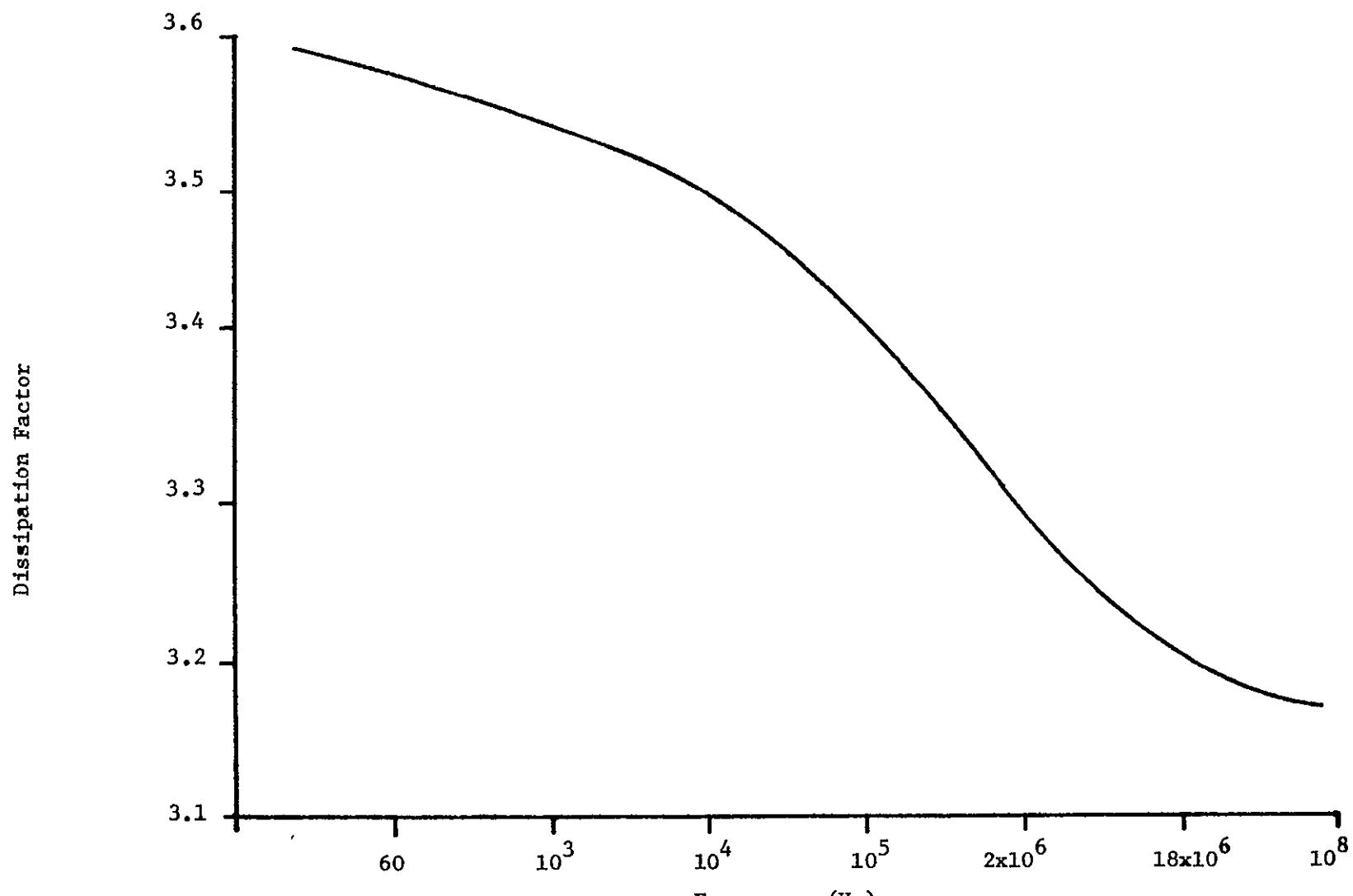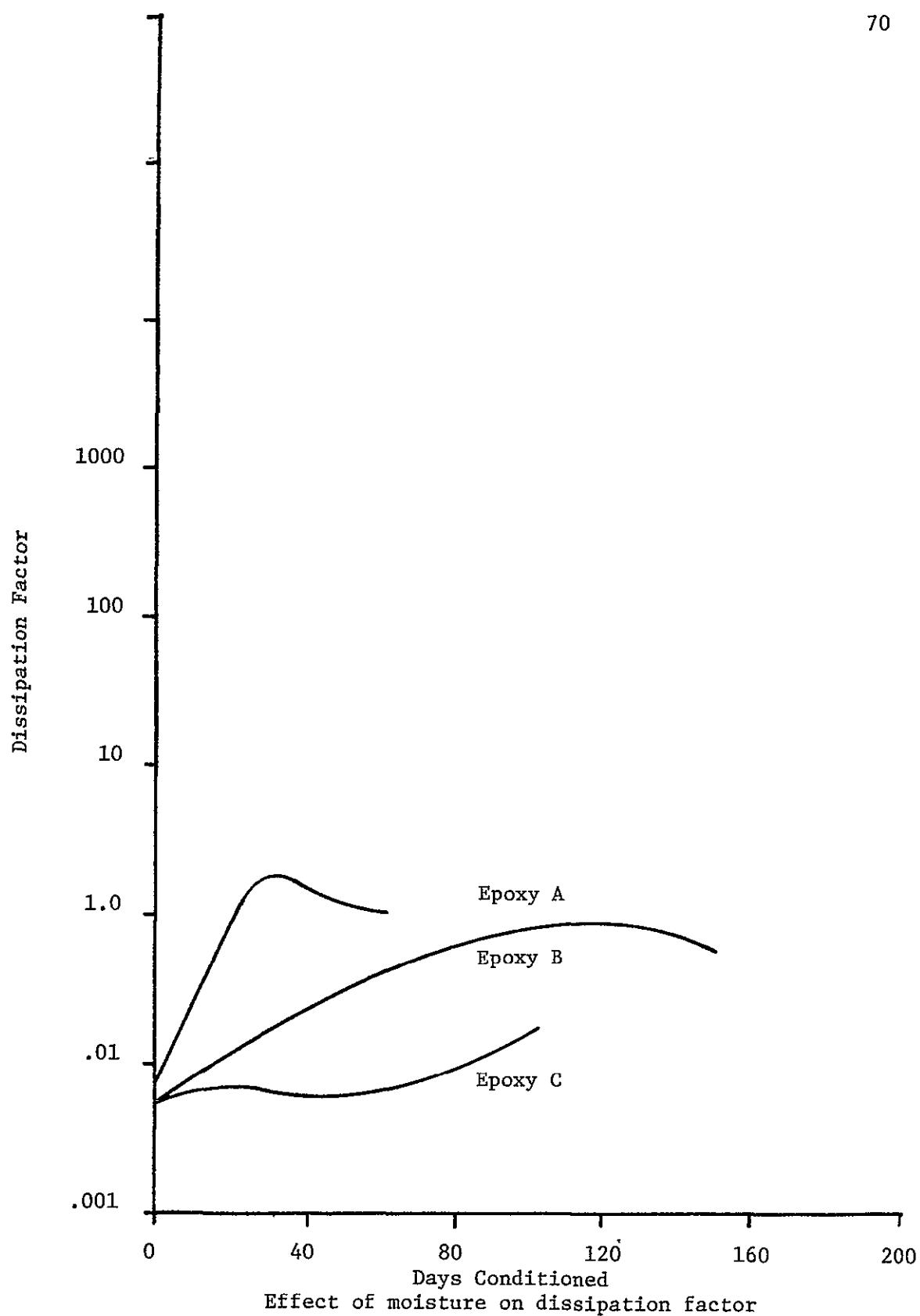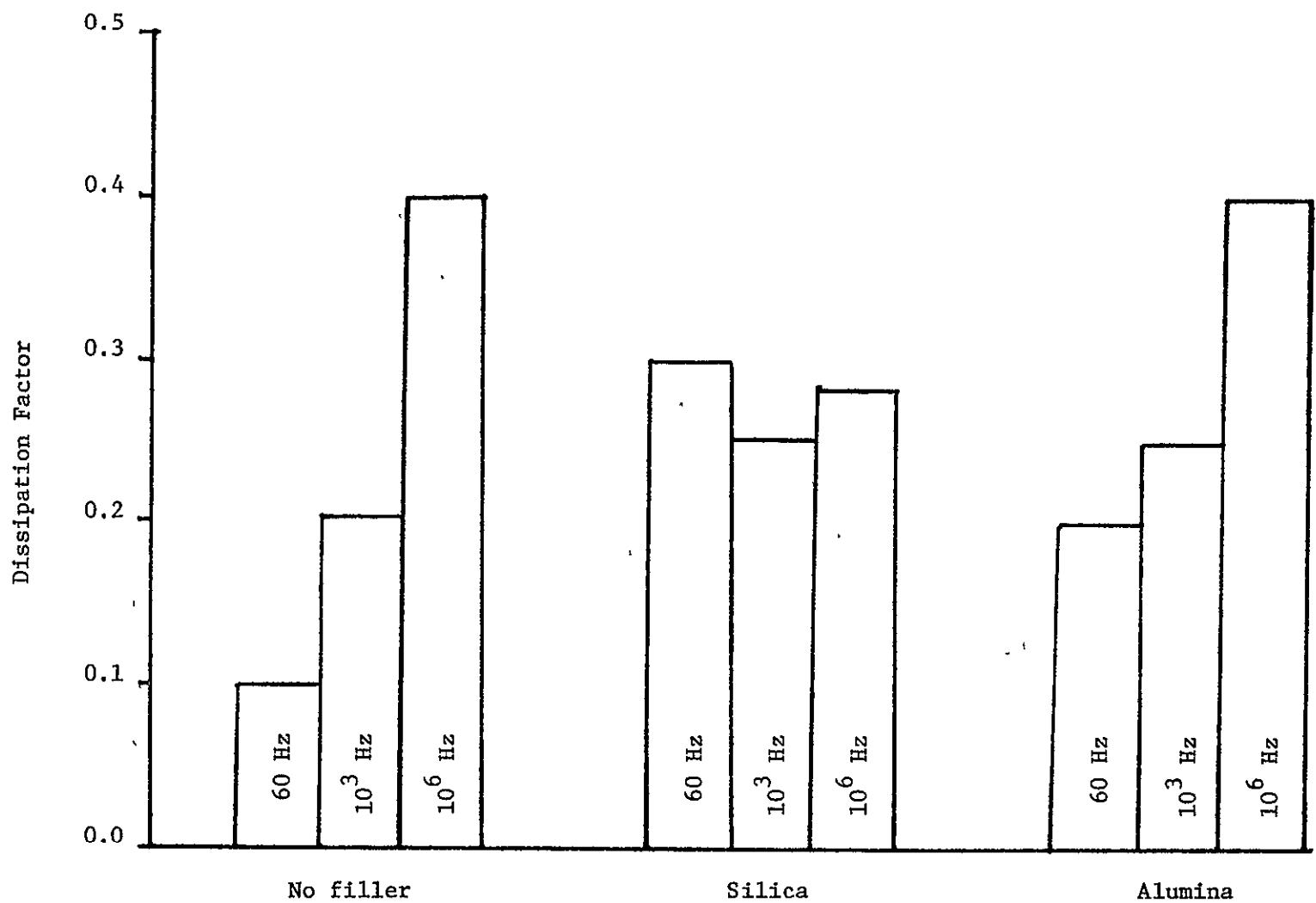
Figure 7.12
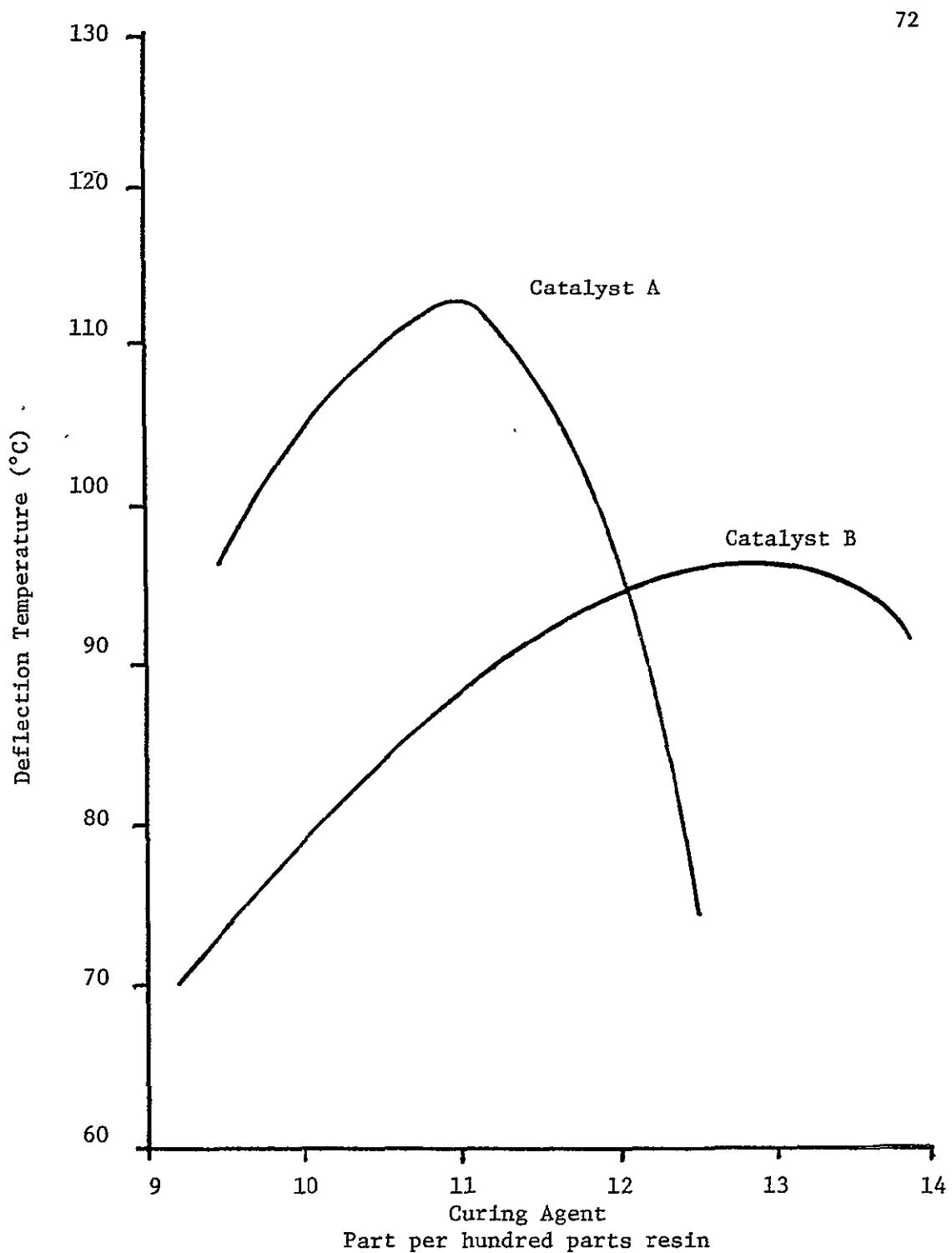
Figure 7.13

Effect of catalyst concentration

Figure 7.14

about lo^{13} ohm-cm. Conductive materials, however, may have resistivities

as low as 10^{-4} ohm-cm. When speaking in terms of conductive materials,

conductivity (the reciprocal of resistivity) is generally used to

describe the ability of the material to conduct electric current. Non-

conductive fillers are used with electrical grade epoxies and result

in slight changes to original characteristics. Metallic fillers may be

used in high concentrations to form conductive epoxies. Even small

amounts, as little as two parts per hundred parts of resin, of metallic

filler will reduce volume resistivity by a factor of ten. Flaked silver

in combination with a silver powder may yield a resistivity of less

than 4×10^{-4} ohm-cm at room temperature. The higher the value of

resistivity the better the adhesive will insulate and, conversely,

the lower the value the better the conduction of current. Resistivity

of an adhesive varies with a number of factors. It varies inversely

with temperature, and is affected by moisture and humidity, the level

of voltage applied, and the length of time the voltage is being

applied. Volume resistivity for a typical unfilled polyimide is shown

in Figure 7.15.[13] Filler loading volume also affects the resistivity

(Fig. 7.16).[14] Nonconductive fillers tend to increase resistivity

and metallic fillers tend to decrease the resistivity as would be

expected.

In hybrid microelectronics, both conductive and nonconductive

epoxies are used. The conductive epoxies are used primarily to attach

active and passive devices to subtrates, but they may also be used to

attach substrates to packages if electrical insulation is not a

problem. Packages and lids may also be sealed with conductive epoxies

for shielding purposes. Gold and silver are primarily fillers for
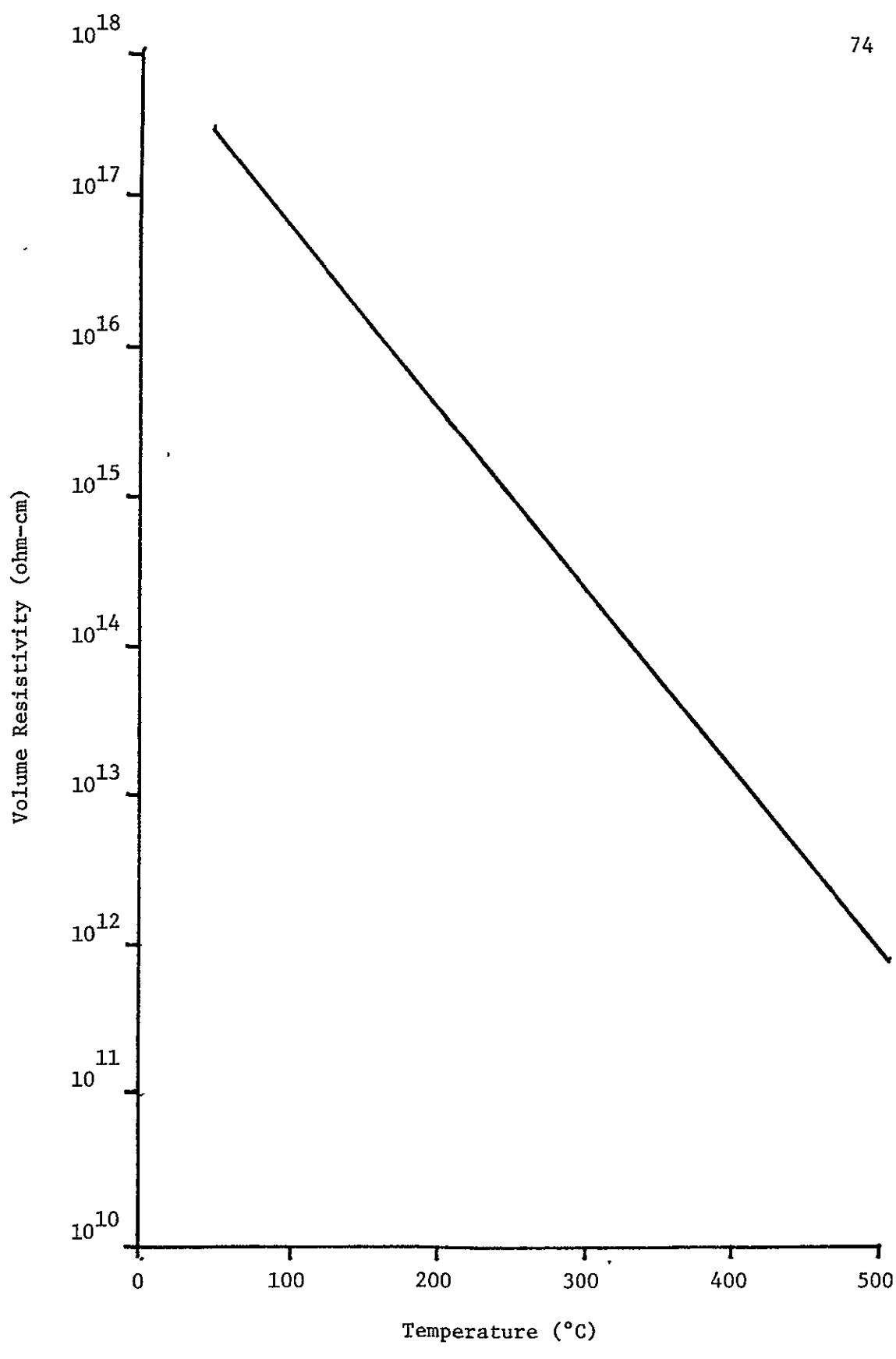
Figure 7.15
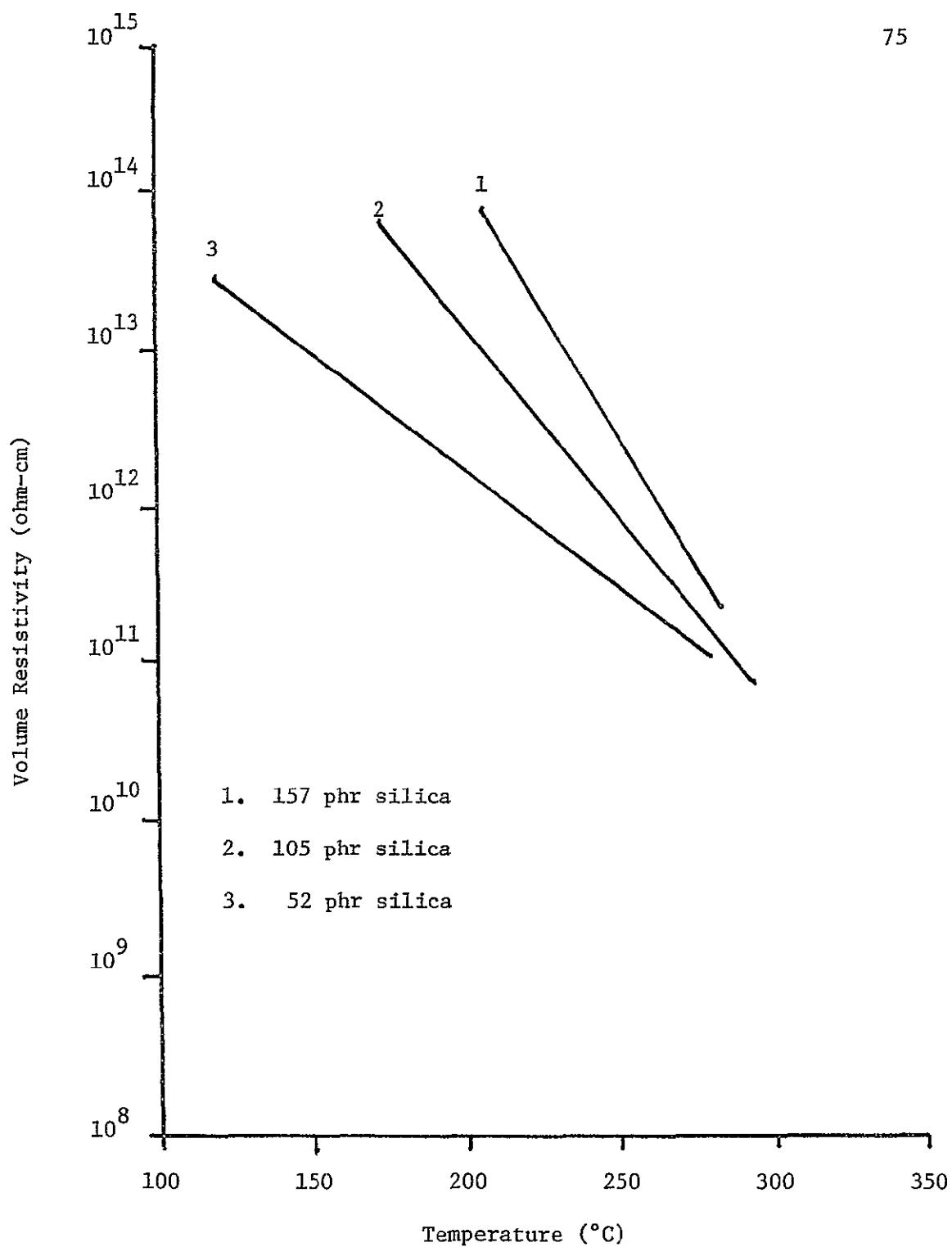
Figure 7.16

conductive epoxies used in microelectronics. However, some epoxies use silver coated glass spheres for fillers instead of silver flakes with only a slight difference in volume resistivity.

Nonconductive epoxies are found to be primarily silica filled or to have no filler at all. In microelectronics, these epoxies are used primarily for chip bonding where no electrical contact in necessary, for hermetic sealing, and in bonding substrates to packages. When filled, these epoxies become excellent thermal conductors. More will be said about conductive and nonconductive epoxies with regards to particular adhesives in Chapter IX.

Footnotes

[1]Charles A. Harper, ed., Handbook of Plastics and Elastomers (New York, McGraw-Hill Book Company, 1975), p. 2.63.

[2]Henry Lee and Kriss Neville, ed., Handbook of Epoxy Resins (New York, McGraw-Hill Book Company, 1967), p. 14.39.

[3]Ibid., p. 6.53.

[4]Ibid., p. 8.3.

[5]Ibid., p. 14.35.

[6]Ibid., pp. 7.5 and 11.7.

[7]Ibid., p. 6.14.

[8]Cree W. Stout, J.E. Sergent, S.V. Caruso, "Electrical Properties of Adhesives Used in Hybrid Microelectronics," Proceedings of the Electronic Components Convention, San Francisco, California, 1976.

[9]Lee, et al., p. 8.14.

[10]Ibid., p. 17.26.

[11]Ibid., p. 14.39.

[12]Ibid., p. 7.7.

[13]Harper, p. 2.63.

[14]Lee, et al., p. 14.34.

Chapter VIII

Measurement Techniques
and
Experimental Results

This chapter deals with techniques for measuring the resistivity

of conductive epoxies and the dielectric properties of nonconductive

epoxies. Studies of these measurement techniques were part of the

requirements specified by NASA contract NAS8-31172. Three techniques

for measuring the resistivity of conductive epoxies and a technique for

measuring the dielectric constant, power factor and loss tangent of

nonconductive epoxies will be discussed. The effect of aging on epoxies

will also be considered.

One technique for measuring resistivity is to use two parallel

strips of scotch tape. This method is being considered by ASTM as

the standard for measuring the resistivity of adhesives. Two thick

film contacts are fabricated on an alumina substrate a known distance

apart. A mold is then formed by placing two double thicknesses of

scotch tape parallel to each other between the thick film contacts.

The mold is then filled with epoxy and leveled with the aid of a

razor blade. The tape is then removed and the epoxy cured as per

manufacturer's recommendations. The dimensions are then measured with

the aid of a light-section microscope, the resistance measured with a

milliohmmeter, and the resistivity calculated by the standard formula:

$$\rho = RA/l$$

where   R = resistance measured,

A = cross sectional area,

1 = length of sample.

This technique was rejected for the following reasons:

1. The samples were not reproducible, making them difficult to compare.

2. The contact resistance between the epoxy and thick film cannot be accounted for. This resistance plus any resistance due to metalization may be erroneously attributed to the epoxy.

3. Test samples take a considerable amount of time to prepare.

4. The equipment needed (light-section microscope, milliohm-meter, etc.) is either expensive or difficult to operate.

Another technique for measuring resistivity is the ac induction method.[1] This method measures resistivities without direct contact to the adhesive, thus eliminating possibilities of surface contact resistance. This is accomplished by introducing an electrical conductor, in the shape of a circular cylinder, into the field of an inductor. In this method, precaution must be taken that the skin depth for the frequency used is equal to, or greater than, the radius of the specimen in order to eliminate confinement of eddy currents to the surface of the sample. In order to use conventional ac circuit theory, Maxwell's equations for fields which vary sinusoidally with time will be solved for the particular solution.

The specimen, for all practical purposes, is a cylinder of circular cross-section made of a conductive epoxy. When this specimen is placed inside an inductor, the impedance measured between the coil terminals at some frequency ω contains both a resistive and a reactive

component in the form of $R + j\omega L$. Letting the resistance R be defined

as the ratio of input power to the square of the rms current (P/I^2), P

is seen to have two components P_1, the resistive loss in the coil, and P_2,

the eddy current loss in the specimen. From P_1 and P_2, corresponding

resistive components R_o and R_1 can be determined such that:

$$R = R_o + R_1.$$

Also E/I^2, where E is the average stored energy, may be defined as the

inductance L, such that:

$$L = L_o + L_1.$$

R_o and L_o are the resistance and inductance respectively of the coil

alone.

The simplest expressions for R_1 and L_1 are for circular cylindrical

geometry (see Appendix). For this case,

$$-\alpha \frac{R_1}{\omega L_o} = \frac{2M_1(x)}{xM_o(x)} \sin [\theta_1(x) - \theta_o(x) - \frac{3\pi}{4}] \qquad \text{(Eq. 8.1)} ,$$

$$-\alpha \frac{L_1}{L_o} = \frac{2M_1(x)}{xM_o(x)} \cos [\theta_1(x) - \theta_o(x) - \frac{3\pi}{4}] - 1 \qquad \text{(Eq. 8.2)}$$

where $x = (\omega \mu_o \sigma)^{1/2} a$

a = radius of cylinder

σ = conductivity of the sample

$\mu_o = 4\pi \times 10^{-7}$ H/m.

$M_o(x)$, $\theta_o(x)$, $M_1(x)$, $\theta_o(x)$ are the polar components of the Bessel

functions $J_o(j^{3/2}x)$ and $J_1(j^{3/2}x)$. For cylindrical geometry, α is

the ratio of the cross-sectional area of the coil to that of the specimen.

Equations 8.1 and 8.2 and their ratio are shown in Figures 8.1a, b, and c.

respectively. The curve in 8.1c is perhaps the most useful since it

does not depend on coil size, shape, or number of turns.

Figure 8.1a

$$\frac{\alpha R_1}{\omega L_o} \times 10^{-1}$$

$(\omega \mu_o \sigma)^{1/2} a$

81a

Figure 8.1b

$$\frac{\alpha L_1}{L_o} \times 10^{-1}$$

$(\omega\mu_o\sigma)^{1/2}a$

Figure 8.1c

$$\frac{\omega L_1}{R_1}$$

$(\omega\mu_o\sigma)^{1/2}a$
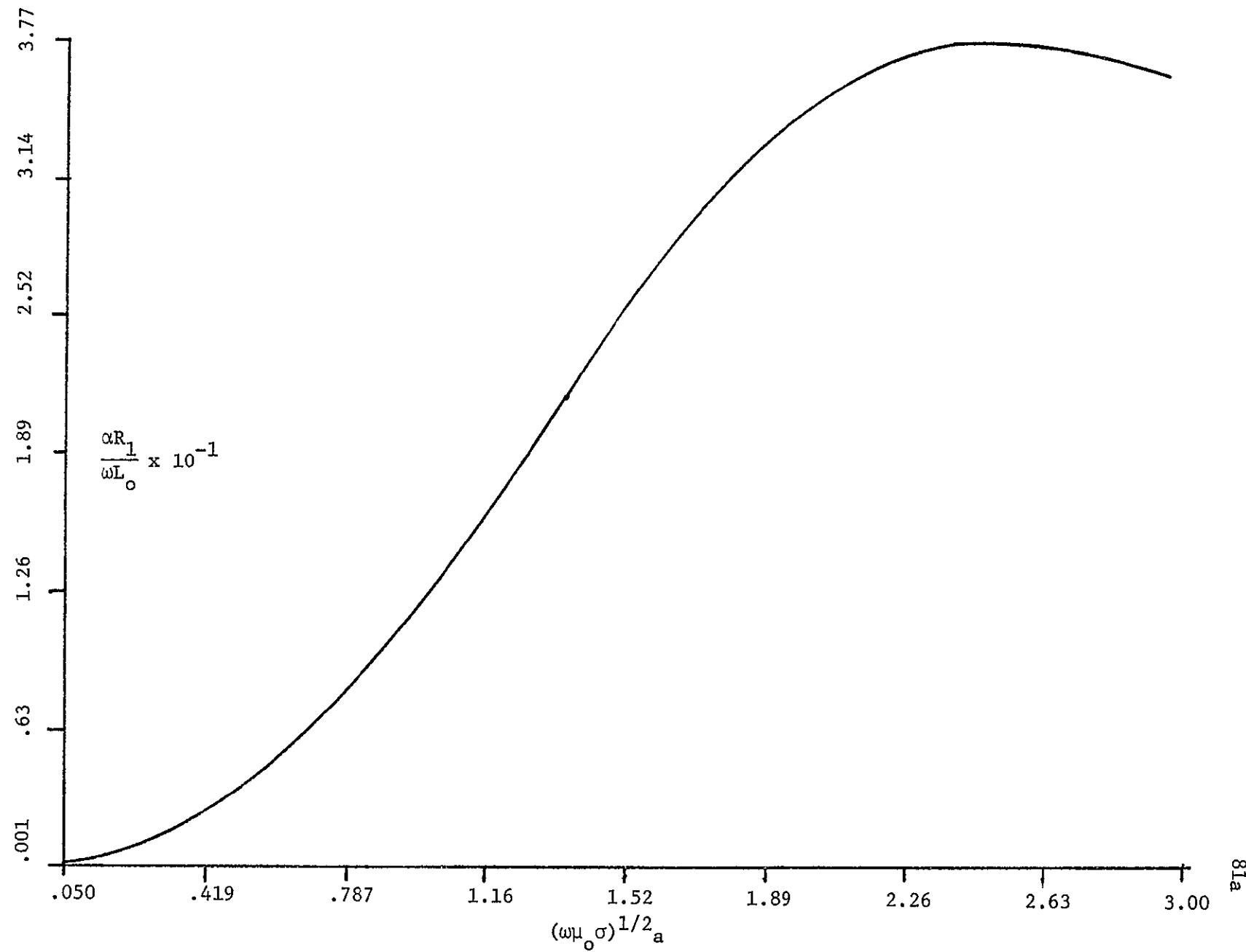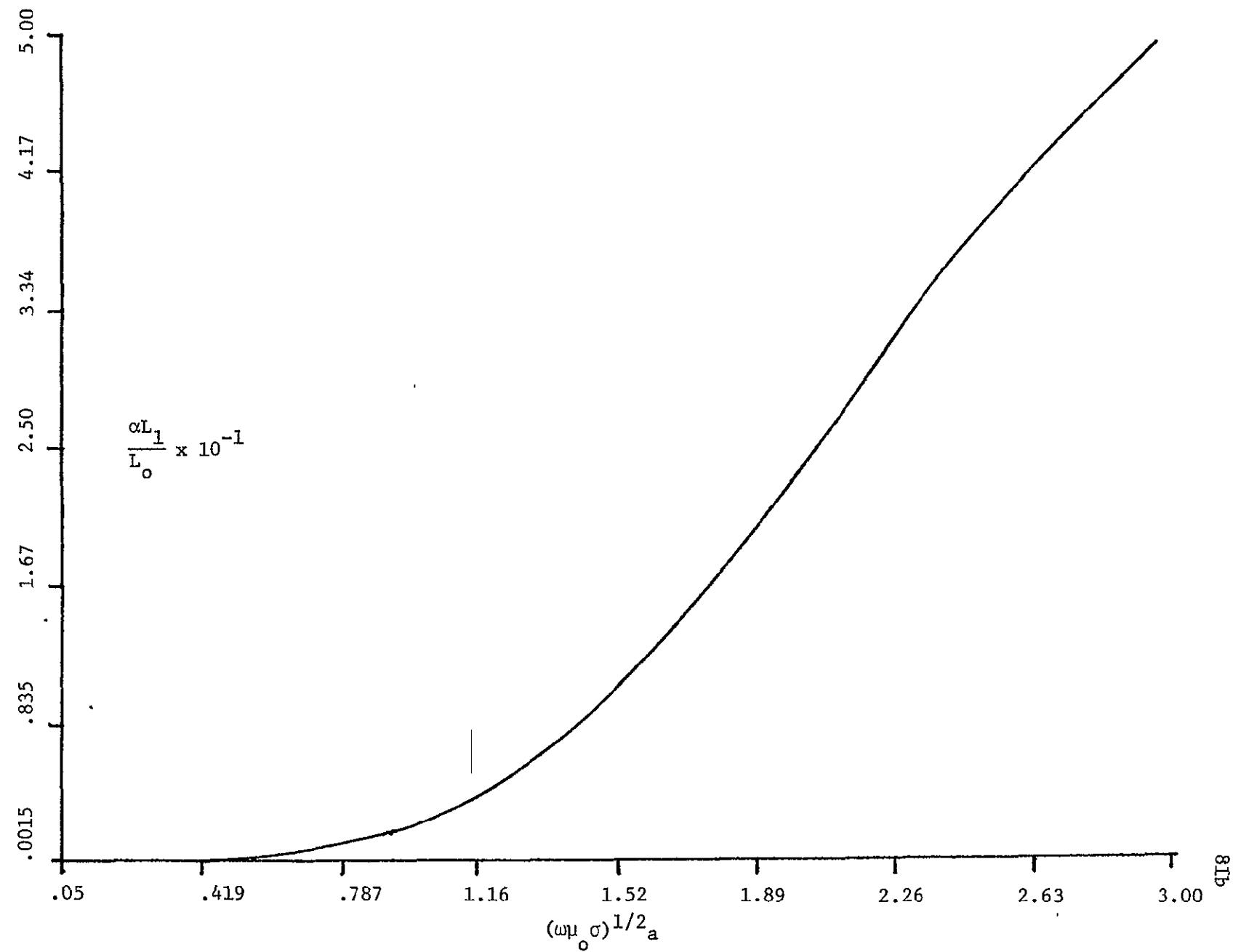
81c
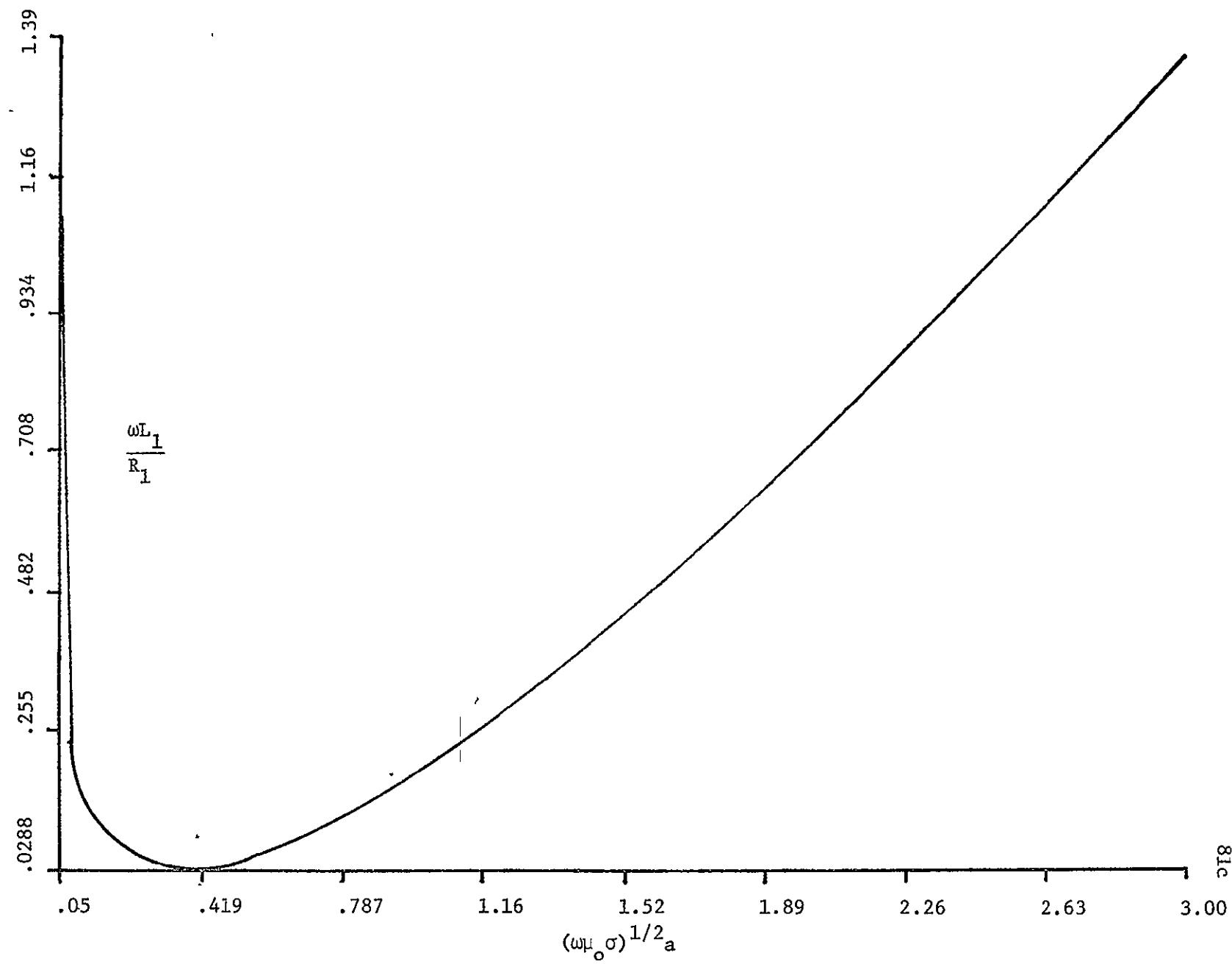
The coil is connected in a Maxwell Bridge configuration as shown in Figure 8.2. Knowing that $\overline{Y}_1\overline{Y}_4 = \overline{Y}_2\overline{Y}_3$ where $\overline{Y}_i$ is the admittance of the i'th arm, the impedance of arm 1 can be calculated by using a modified version of the equation:

$$\overline{Y}_1 = \frac{1}{R_1} + j\omega C_1. \qquad \text{(Eq. 8.3)}$$

If
$$\overline{Z}_2 = R_2 \qquad \text{(Eq. 8.4)}$$

$$\overline{Z}_3 = R_3 \qquad \text{(Eq. 8.5)}$$

$$\overline{Z}_x = R_x + j\omega L_x \qquad \text{(Eq. 8.6)}$$

where
$$\overline{Z}_i = \frac{1}{\overline{Y}_i},$$

substitution into:

$$\overline{Z}_x = \overline{Z}_2\overline{Z}_3\overline{Y}_1$$

yields

$$R_x + j\omega L_x = R_2 R_3 \ [(1/R_1) + j\omega C_1].$$

Separating real and imaginary parts, this becomes:

$$L_x = R_2 R_3 C_1 \qquad \text{Henrys} \qquad \text{(Eq. 8.7)}$$

$$R_x = R_2 R_3 / R_1 \qquad \text{Ohms.} \qquad \text{(Eq. 8.8)}$$

The bridge is balanced with the empty coil and the values are recorded. L_o and R_o are found by substituting measured values into Equations 8.7 and 8.8. The sample is then placed in the coil, and the bridge is again balanced. The values determined here are substituted into Equations 8.7 and 8.8 to solve for L and R respectively. R_1 and L_1 are then determined by Equations 8.1 and 8.2 or from Figures 8.1a, b, or c.

The sample for this system was a circular cross-section cylinder of silver filled epoxy with a radius of 0.56 cm and a length of 2.34 cm.
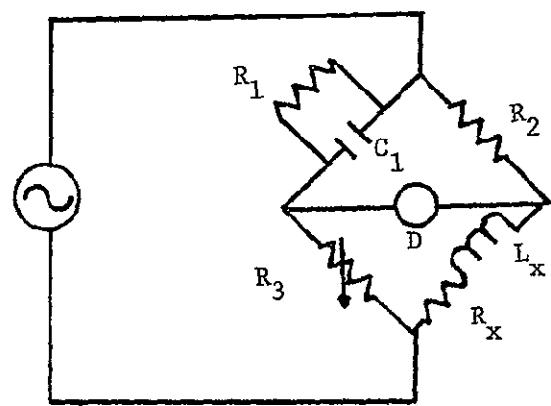
Maxwell Bridge

Figure 8.2

The coil had an inside diameter of 0.57 cm and a length of 2.54 cm. Table 8.1 shows the conductivity of a typical silver filled epoxy found by this method.

For pure silver a skin depth equal to the radius of the sample occurs at a frequency of approximately 500 Hz. Above this the apparent conductivity decreases due to nonuniform field distribution. For samples of epoxy filled with powdered silver, no significant deviation in conductivity occured at less than 5 kHz, indicating that the conductivity of silver filled epoxy is approximately two orders of magnitude less than that of pure silver, a phenomenon also indicated by data taken at dc.

This method yields excellent results for frequencies up to 200MHz. For frequencies greater than this, the system depends on the accuracy of the bridge used. However, this method has some difficulties in obtaining good results. One problem that arises is that the amount of epoxy to be used is considerable, therefore very expensive. Another problem is the difficulty in getting perfectly cyclindrical specimens without air pockets. Students at the University of South Florida are now pursuing other means of measuring resistivities at high frequencies.

A third technique of measuring resistivity is a modified form of the conventional four-point system. This technique requires that the resistivity not depend on the thickness of the adhesive to be measured. Referring to Figure 8.3, a constant current source is connected across a sample of known dimensions to points "a" and "b", generating a voltage across "c" and "d" allowing the resistance of the material to be calculated. The resistance R is given by:

$$R = V/I,$$

Table 8.1

Conductivity of a Typical Silver Filled Epoxy

Frequency	Conductivity
50 Hz	$4.21 \times 10^4 \, \mho/cm$
100 Hz	$4.19 \times 10^4 \, \mho/cm$
500 Hz	$4.16 \times 10^4 \, \mho/cm$
1000 Hz	$4.12 \times 10^4 \, \mho/cm$
5000 Hz	$3.84 \times 10^4 \, \mho/cm$
10000 Hz	$3.53 \times 10^4 \, \mho/cm$
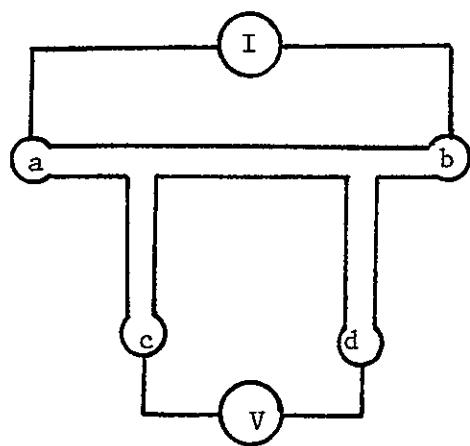
Four-Point System

Figure 8.3

where   V = voltage generated

    I = applied current.

The resistivity can be found from this equation and the known dimensions
of the specimen:

$$\rho = RA/l \qquad\qquad\text{(Eq. 8.9)}$$

where   A = wt the cross-sectional area

    l = length of sample

    w = width of sample

    t = thickness of sample.

From Equation 8.9, the conductivity can be calculated as

$$\sigma = 1/\rho. \qquad\qquad\text{(Eq. 8.10)}$$

For this system, a mold was machined in Rexolite, a very high quali-
ty dielectric material, in the shape and dimensions shown in Figure 8.4.
The mold is then filled with epoxy and cured per manufacturer's recom-
mendations. All testing is then completed with the epoxy remaining in
the mold. The mold of cured epoxy is placed in a test fixture designed
as shown in Figure 8.5. Resistivity was calculated as previously stated
over a range of applied current from 0.1 to 0.5 amps in one-tenth amp
intervals. Tables 8.2 through 8.37 show some average values of resisti-
vities for various conductive epoxies.

Rexolite was chosen, not only because of its high quality dielectric
properties, but also because it can be easily machined to a high degree
of precision and tolerance. Rexolite also maintains its dimensional
integrity over a wide temperature range. Thus curing and temperature
storage will not affect the dimensions of the epoxy sample. Since
Rexolite is very machinable, the molds may be made any size and depth
desired.
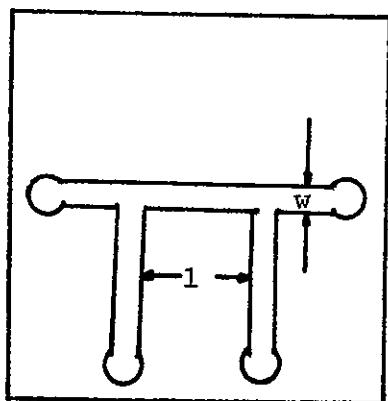
Mold Configuration

Figure 8.4

Table 8.2

Resistivity of Epo-Tec H11

Filler: Silver		2 Components
Temperature (°C)	Resistivity (Ω-cm)	
-55	2.32×10^{-4}	
-20	2.52×10^{-4}	
0	2.71×10^{-4}	
25	$2.92 \times .0^{-4}$	
60	3.22×10^{-4}	
100	3.57×10^{-4}	
150	4.18×10^{-4}	

Temperature Coefficient of Resistance:

-55° to 0°C   3056 ppm/°C

0° to 150°C   3616 ppm/°C

Table 8.3

Epo-Tec H11

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	3.10×10^{-4}
	0.2	3.11×10^{-4}
	0.3	3.09×10^{-4}
	0.4	3.09×10^{-4}
	0.5	3.09×10^{-4}
500	0.1	3.77×10^{-4}
	0.2	3.73×10^{-4}
	0.3	3.70×10^{-4}
	0.4	3.68×10^{-4}
	0.5	3.69×10^{-4}
1000	0.1	4.02×10^{-4}
	0.2	4.03×10^{-4}
	0.3	3.99×10^{-4}
	0.4	3.95×10^{-4}
	0.5	3.95×10^{-4}

Table 8.4

Resistivity of Epo-Tec H20E

Filler: Silver		2 Components
Temperature (°C)		Resistivity (Ω-cm)
−55		1.25×10^{-4}
−20		1.39×10^{-4}
0		1.45×10^{-4}
25		1.55×10^{-4}
60		1.69×10^{-4}
100		1.88×10^{-4}
150		1.90×10^{-4}

Temperature Coefficient of Resistance:

−55° to 0°C   2909 ppm/°C

0° to 150°C   2069 ppm/°C

Table 8.5

Epo-Tec H20E

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	1.64×10^{-4}
	0.2	1.61×10^{-4}
	0.3	1.59×10^{-4}
	0.4	1.56×10^{-4}
	0.5	1.55×10^{-4}
500	0.1	1.60×10^{-4}
	0.2	1.56×10^{-4}
	0.3	1.55×10^{-4}
	0.4	1.54×10^{-4}
	0.5	1.54×10^{-4}
1000	0.1	1.54×10^{-4}
	0.2	1.51×10^{-4}
	0.3	1.50×10^{-4}
	0.4	1.50×10^{-4}
	0.5	1.50×10^{-4}

Table 8.6

Epo-Tec H20E

Sample "A"

Resistivity versus Frequency

Frequency (Hz)	Resistivity (Ω-cm)
dc	3.57×10^{-4}
100	3.09×10^{-4}
200	3.09×10^{-4}
500	3.09×10^{-4}
1,000	3.09×10^{-4}
2,000	3.09×10^{-4}
5,000	3.12×10^{-4}
10,000	3.15×10^{-4}
20,000	3.18×10^{-4}
50,000	3.18×10^{-4}
100,000	3.18×10^{-4}
200,000	3.25×10^{-4}

Table 8.7

Epo—Tec H20E

Sample "B"

Resistivity versus Frequency

Frequency (Hz)	Resistivity (Ω-cm)
dc	3.37×10^{-4}
100	3.38×10^{-4}
200	3.38×10^{-4}
500	3.38×10^{-4}
1,000	3.38×10^{-4}
2,000	3.40×10^{-4}
5,000	3.43×10^{-4}
10,000	3.45×10^{-4}
20,000	3.45×10^{-4}
50,000	3.45×10^{-4}
100,000	3.43×10^{-4}
200,000	3.43×10^{-4}

Table 8.8

Resistivity of Epo-Tec H21D

Filler: Silver		2 Components
Temperature (°C)	-	Resistivity (Ω-cm) .
-55		5.27×10^{-4}
-20		5.73×10^{-4}
0		6.03×10^{-4}
25		6.51×10^{-4}
60		7.03×10^{-4}
100		7.82×10^{-4}
150		8.59×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   2622 ppm/°C

0° to 150°C   2830 ppm/°C

Table 8.9

Epo-Tec H21D

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	7.47×10^{-4}
	0.2	7.36×10^{-4}
	0.3	7.32×10^{-4}
	0.4	7.31×10^{-4}
	0.5	7.28×10^{-4}
500	0.1	7.32×10^{-4}
	0.2	7.15×10^{-4}
	0.3	7.08×10^{-4}
	0.4	7.06×10^{-4}
	0.5	7.06×10^{-4}
1000	0.1	7.12×10^{-4}
	0.2	6.95×10^{-4}
	0.3	6.99×10^{-4}
	0.4	6.93×10^{-4}
	0.5	6.97×10^{-4}

Table 8.10

Resistivity of Epo-Tec H31

Filler: Silver	1 Components
Temperature (°C)	Resistivity (Ω-cm)
-55	3.13×10^{-4}
-20	3.49×10^{-4}
0	3.84×10^{-4}
25	4.07×10^{-4}
60	4.33×10^{-4}
100	4.94×10^{-4}
150	5.18×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   4124 ppm/°C

0° to 150°C   2326 ppm/°C

Table 8.11

Epo-Tec H31

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	3.10×10^{-4}
	0.2	3.11×10^{-4}
	0.3	3.09×10^{-4}
	0.4	3.09×10^{-4}
	0.5	3.09×10^{-4}
500	0.1	3.77×10^{-4}
	0.2	3.73×10^{-4}
	0.3	3.70×10^{-4}
	0.4	3.68×10^{-4}
	0.5	3.69×10^{-4}
1000	0.1	4.02×10^{-4}
	0.2	4.03×10^{-4}
	0.3	3.99×10^{-4}
	0.4	3.95×10^{-4}
	0.5	3.95×10^{-4}

Table 8.12

Resistivity of Epo-Tec H31D

Filler: __Silver__ __1__ Components

Temperature (°C)	Resistivity (Ω-cm)
-55	2.72×10^{-4}
-20	2.93×10^{-4}
0	3.12×10^{-4}
25	3.35×10^{-4}
60	3.68×10^{-4}
100	4.04×10^{-4}
150	4.47×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   2694 ppm/°C

0° to 150°C   2863 ppm/°C

Table 8.13

Epo-Tec H31D

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	3.82×10^{-4}
	0.2	3.74×10^{-4}
	0.4	3.74×10^{-4}
	0.5	3.74×10^{-4}
500	0.1	3.96×10^{-4}
	0.2	3.81×10^{-4}
	0.3	3.81×10^{-4}
	0.4	3.81×10^{-4}
	0.5	3.81×10^{-4}
1000	0.1	3.92×10^{-4}
	0.2	3.74×10^{-4}
	0.3	3.79×10^{-4}
	0.4	3.74×10^{-4}
	0.5	3.74×10^{-4}

Table 8.14

Resistivity of Epo-Tec H31LV

Filler: Silver	1 Components
Temperature (°C)	Resistivity (Ω-cm)
-55	6.71×10^{-4}
-20	7.54×10^{-4}
0	8.02×10^{-4}
25	8.53×10^{-4}
60	9.53×10^{-4}
100	10.94×10^{-4}
150	9.35×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°c   3528 ppm/°C

  0° to 150°C   3098 ppm/°C

Table 8.15

Epo-Tec H31LV

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	10.236×10^{-3}
	0.2	10.049×10^{-3}
	0.3	10.153×10^{-3}
	0.4	10.439×10^{-3}
	0.5	10.759×10^{-3}
500	0.1	14.18×10^{-3}
	0.2	14.44×10^{-3}
	0.3	14.94×10^{-3}
	0.4	15.74×10^{-3}
	0.5	15.65×10^{-3}
1000	0.1	13.83×10^{-3}
	0.2	14.33×10^{-3}
	0.3	16.07×10^{-3}
	0.4	16.71×10^{-3}
	0.5	16.93×10^{-3}

Table 8.16

Resistivity of Epo-Tec H40

Filler: <u>Gold</u>   <u>1</u>   Components

Temperature (°C)	Resistivity (Ω-cm)
-55	5.16×10^{-4}
-20	5.71×10^{-4}
0	6.11×10^{-4}
25	6.73×10^{-4}
60	7.99×10^{-4}
100	9.50×10^{-4}
150	12.59×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   3347 ppm/°C

0° to 150°C   7070 ppm/°C

Table 8.17

Epo-Tec H40

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	6.77×10^{-4}
	0.2	6.52×10^{-4}
	0.3	6.54×10^{-4}
	0.4	6.53×10^{-4}
	0.5	6.57×10^{-4}
500	0.1	6.96×10^{-4}
	0.2	6.90×10^{-4}
	0.3	6.90×10^{-4}
	0.4	6.91×10^{-4}
	0.5	6.95×10^{-4}
1000	0.1	6.89×10^{-4}
	0.2	6.90×10^{-4}
	0.3	6.87×10^{-4}
	0.4	6.88×10^{-4}
	0.5	6.87×10^{-4}

Table 8.18

Resistivity of Epo-Tec H41

Filler: <u>Gold</u>   <u>1</u>   Componesnts

Temperature (°C)	Resistivity (Ω-cm)
-55	3.94×10^{-4}
-20	4.35×10^{-4}
0	4.65×10^{-4}
25	5.12×10^{-4}
60	6.18×10^{-4}
100	8.12×10^{-4}
150	11.18×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   3276 ppm/°C

0° to 150°C   9362 ppm/°C

Table 8.19

Epo-Tec H41

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm–cm)
No aging	0.1	4.90×10^{-4}
	0.2	4.85×10^{-4}
	0.3	4.81×10^{-4}
	0.4	4.81×10^{-4}
	0:5	4.84×10^{-4}
500	0.1	4.92×10^{-4}
	0.2	4.94×10^{-4}
	0.3	4.94×10^{-4}
	0.4	4.90×10^{-4}
	0.5	4.94×10^{-4}
1000	0.1	4.90×10^{-4}
	0.2	4.84×10^{-4}
	0.3	4.83×10^{-4}
	0.4	4.79×10^{-4}
	0.5	4.82×10^{-4}

Table 8.20

Resistivity of Epo-Tec H43

Temperature (°C)	Resistivity (Ω-cm)
-55	4.88×10^{-4}
-20	5.29×10^{-4}
0	5.70×10^{-4}
25	6.08×10^{-4}
60	6.72×10^{-4}
100	7.60×10^{-4}
150	9.09×10^{-4}

Temperature Coefficient of Resistanct:

-55° to 0°C   3055 ppm/°C

  0° to 150°C 3965 ppm/°C

Table 8.21

Epo-Tec H43

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	$.5.84 \times 10^{-4}$
	0.2	5.84×10^{-4}
	0.3	5.75×10^{-4}
	0.4	5.73×10^{-4}
	0.5	5.75×10^{-4}
500	0.1	5.43×10^{-4}
	0.2	5.42×10^{-4}
	0.3	5.49×10^{-4}
	0.4	5.50×10^{-4}
	0.5	5.48×10^{-4}
1000	0.1	5.42×10^{-4}
	0.2	5.36×10^{-4}
	0.3	5.36×10^{-4}
	0.4	5.35×10^{-4}
	0.5	5.37×10^{-4}

Table 8.22

Resistivity of Epo-Tec H44

Filler: <u>Gold</u>	<u>1</u> Components
Temperature (°C)	Resistivity (Ω-cm)
-55	7.15×10^{-4}
-20	7.45×10^{-4}
0	8.00×10^{-4}
25	8.58×10^{-4}
60	9.27×10^{-4}
100	10.28×10^{-4}
150	11.45×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   2165 ppm/°C

 0° to 150°C   2879 ppm/°C

Table 8.23

Epo-Tec H44

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	9.05×10^{-4}
	0.2	8.96×10^{-4}
	0.3	9.02×10^{-4}
	0.4	8.92×10^{-4}
	0.5	8.96×10^{-4}
500	0.1	9.01×10^{-4}
	0.2	9.40×10^{-4}
	0.3	8.77×10^{-4}
	0.4	8.72×10^{-4}
	0.5	8.73×10^{-4}
1000	0.1	8.85×10^{-4}
	0.2	8.68×10^{-4}
	0.3	8.62×10^{-4}
	0.4	8.68×10^{-4}
	0.5	8.67×10^{-4}

Table 8.24

Resistivity of Epo-Tec H81

Filler: <u>Gold</u>	<u>2</u> Components
Temperature (°C)	Resistivity (Ω-cm)
-55	9.39×10^{-4}
-20	10.48×10^{-4}
0	10.97×10^{-4}
25	11.69×10^{-4}
60	12.91×10^{-4}
100	14.32×10^{-4}
150	16.21×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   3062 ppm/°C

 0° to 150°C   3188 ppm/°C

Table 8.25

Epo-Tec H81

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	12.75×10^{-4}
	0.2	12.54×10^{-4}
	0.3	12.53×10^{-4}
	0.4	12.60×10^{-4}
	0.5	12.56×10^{-4}
500	0.1	11.64×10^{-4}
	0.2	11.78×10^{-4}
	0.3	11.81×10^{-4}
	0.4	11.76×10^{-4}
	0.5	11.88×10^{-4}
1000	0.1	11.74×10^{-4}
	0.2	11.57×10^{-4}
	0.3	11.57×10^{-4}
	0.4	11.62×10^{-4}
	0.5	11.69×10^{-4}

Table 8.26

Resistivity of Epo-Tec 410

Filler: Silver		2 Components
	Temperature (°C)	Resistivity (Ω-cm)
	-55	3.47×10^{-4}
	-20	3.93×10^{-4}
	0	4.19×10^{-4}
	25	4.55×10^{-4}
	60	5.01×10^{-4}
	100	5.64×10^{-4}
	150	6.71×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   3747 ppm/°C

0° to 150°C   3992 ppm/°C

Table 8.27

Epo-Tec 410

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	4.52×10^{-4}
	0.2	4.45×10^{-4}
	0.3	4.49×10^{-4}
	0.4	4.46×10^{-4}
	0.5	4.47×10^{-4}
500	0.1	5.28×10^{-4}
	0.2	5.09×10^{-4}
	0.3	5.14×10^{-4}
	0.4	5.11×10^{-4}
	0.5	5.13×10^{-4}
1000	0.1	5.41×10^{-4}
	0.2	5.21×10^{-4}
	0.3	5.19×10^{-4}
	0.4	5.17×10^{-4}
	0.5	5.19×10^{-4}

Table 8.28

Resistivity of Epo-Tec 410E

Filler: Silver	2 Components
Temperature (°C)	Resistivity (Ω-cm)
-55	2.33×10^{-4}
-20	2.65×10^{-4}
0	2.85×10^{-4}
25	3.09×10^{-4}
60	3.45×10^{-4}
100	3.84×10^{-4}
150	4.40×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   4029 ppm/°C

0° to 150°C   3628 ppm/°C

Table 8.29

Epo-Tec 410E

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	3.50×10^{-4}
	0.2	3.50×10^{-4}
	0.3	3.45×10^{-4}
	0.4	3.44×10^{-4}
	0.5	3.44×10^{-4}
500	0.1	4.13×10^{-4}
	0.2	4.07×10^{-4}
	0.3	4.02×10^{-4}
	0.4	4.02×10^{-4}
	0.5	4.01×10^{-4}
1000	0.1	4.06×10^{-4}
	0.2	4.04×10^{-4}
	0.3	4.02×10^{-4}
	0.4	4.02×10^{-4}
	0.5	4.02×10^{-4}

Table 8.30

Resistivity of Epo-Tec 410LV

Filler: Silver	2 Components
Temperature (°C)	Resistivity (Ω-cm)
−55	1.51×10^{-3}
−20	1.65×10^{-3}
0	1.83×10^{-3}
25	1.98×10^{-3}
60	2.19×10^{-3}
100	2.52×10^{-3}
150	3.22×10^{-3}

Temperature Coefficient of Resistance:

−55° to 0°C   3788 ppm/°C

0° to 150°C 5103 ppm/°C

Table 8.31

Epo-Tec 410LV

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	19.65×10^{-4}
	0.2	19.53×10^{-4}
	0.3	19.53×10^{-4}
	0.4	19.53×10^{-4}
	0.5	19.78×10^{-4}
500	0.1	31.49×10^{-4}
	0.2	30.86×10^{-4}
	0.3	30.60×10^{-4}
	0.4	30.72×10^{-4}
	0.5	31.24×10^{-4}
1000	0.1	31.85×10^{-4}
	0.2	31.44×10^{-4}
	0.3	31.29×10^{-4}
	0.4	31.74×10^{-4}
	0.5	31.65×10^{-4}

Table 8.32

Resistivity of Epo-Tec 417

Filler: Silver	2 Components
Temperature (°C)	Resistivity (Ω-cm)
-55	1.40×10^{-4}
-20	1.58×10^{-4}
0	1.69×10^{-4}
25	1.83×10^{-4}
60	2.02×10^{-4}
100	2.23×10^{-4}
150	2.57×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   3767 ppm/°C

0° to 150°C   3480 ppm/°C

Table 8.33

Epo-Tec 417

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	1.86×10^{-4}
	0.2	1.85×10^{-4}
	0.3	1.85×10^{-4}
	0.4	1.85×10^{-4}
	0.5	1.84×10^{-4}
500	0.1	2.24×10^{-4}
	0.2	2.21×10^{-4}
	0.3	2.19×10^{-4}
	0.4	2.18×10^{-4}
	0.5	2.20×10^{-4}
1000	0.1	2.24×10^{-4}
	0.2	2.23×10^{-4}
	0.3	2.22×10^{-4}
	0.4	2.21×10^{-4}
	0.5	2.21×10^{-4}

Table 8.34

Resistivity of Ablebond 36-2

Filler: Silver	1 Component
Temperature (°C)	Resistivity (Ω-cm)
-55	1.36×10^{-4}
-20	1.47×10^{-4}
0	1.55×10^{-4}
25	1.67×10^{-4}
60	1.81×10^{-4}
100	2.00×10^{-4}
150	2.09×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   2525 ppm/°C

  0° to 150°C   2331 ppm/°C

Table 8.35

Ablebond 36-2

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	1.66×10^{-4}
	0.2	1.63×10^{-4}
	0.3	1.60×10^{-4}
	0.4	1.60×10^{-4}
	0.5	1.59×10^{-4}
500	0.1	1.65×10^{-4}
	0.2	1.61×10^{-4}
	0.3	1.59×10^{-4}
	0.4	1.58×10^{-4}
	0.5	1.58×10^{-4}
1000	0.1	1.54×10^{-4}
	0.2	1.55×10^{-4}
	0.3	1.55×10^{-4}
	0.4	1.54×10^{-4}
	0.5	1.54×10^{-4}

Table 8.36

Resistivity of Ablebond 58-1

Filler: <u>Gold</u>	<u>1</u> Component
Temperature (°C)	Resistivity (Ω-cm)
-55	5.29×10^{-4}
-20	5.33×10^{-4}
0	5.72×10^{-4}
25	6.26×10^{-4}
60	6.67×10^{-4}
100	7.53×10^{-4}
150	8.98×10^{-4}

Temperature Coefficient of Resistance:

-55° to 0°C   1472 ppm/°C

0° to 150°C   3803 ppm/°C

Table 8.37

Ablebond 58-1

Aging Effect on Resistivity

Aging Time (hours)	Current (amps)	Resistivity (ohm-cm)
No aging	0.1	6.49×10^{-4}
	0.2	6.44×10^{-4}
	0.3	6.36×10^{-4}
	0.4	6.33×10^{-4}
	0.5	6.26×10^{-4}
500	0.1	6.36×10^{-4}
	0.2	6.20×10^{-4}
	0.3	6.34×10^{-4}
	0.4	6.30×10^{-4}
	0.5	6.24×10^{-4}
1000	0.1	6.14×10^{-4}
	0.2	6.05×10^{-4}
	0.3	6.06×10^{-4}
	0.4	6.02×10^{-4}
	0.5	6.04×10^{-4}
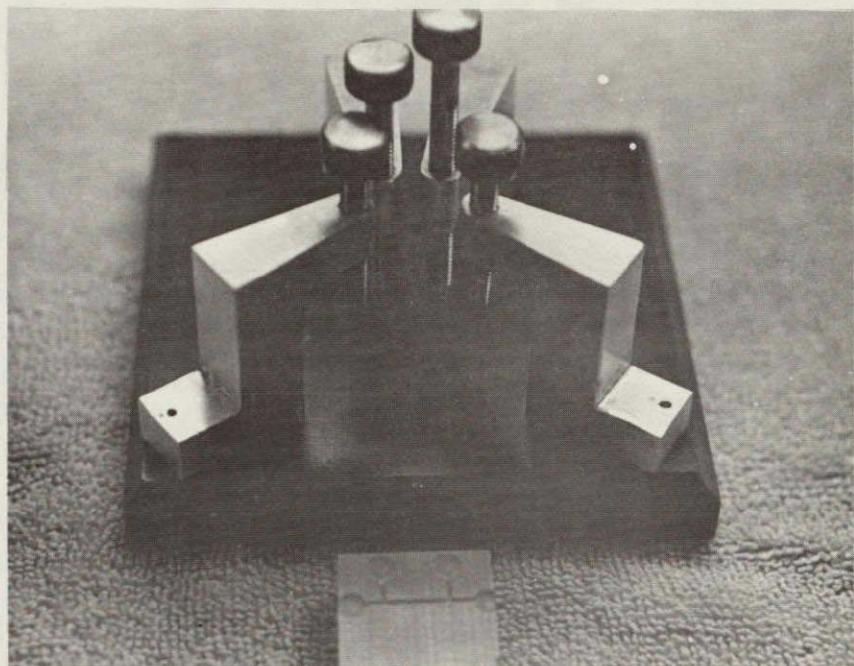
Four-Point System

Test Set

Figure 8.5

This four-point system was also used to measure resistivities up to 50 kHz. Above this frequency, the skin depth began to affect the resistivity greatly. For this method a constant ac current source, varying only with frequency, was applied to points "a" and "b" and the voltage measured between "c" and "d". The experimental samples were the same as those used for the dc testing. Tables 8.6 and 8.7 show data taken in this manner for a silver epoxy. Aging only slightly affected most of the epoxies studied as indicated in Tables 8.2 through 8.37.

The techniques for measuring the dielectric constant, loss tangent, and power factor of nonconductive epoxies makes use of a variable capacitor constructed as shown in Figure 8.6.[2] A flat specimen, large enough to project well over the guarded electrode (preferably well outside the guard electrode), is placed between the moving electrode and the guarded electrode. The micrometer is adjusted such that the moving electrode is moved downward toward the specimen until only a very slight air gap remains between the electrode and the specimen; the micrometer is then read and noted as t_1. The leads of the variable capacitor are connected to a capacitance bridge and the bridge nulled. The specimen is then removed and the variable capacitor adjusted until the bridge is again nulled and the reading, t_2, is noted. The distance the electrode has moved is recorded as:

$$t_x = t_1 - t_2 \quad cm.$$

The relative dielectric constant can then be found by:

$$\varepsilon_r = \frac{t_s}{t_s - t_x}$$

where t_s = thickness of the sample in cm.

Precision
Micrometer

Moving Electrode

Ground

Guard
Ring

Guarded Electrode

Dielectric Properties
Test Set
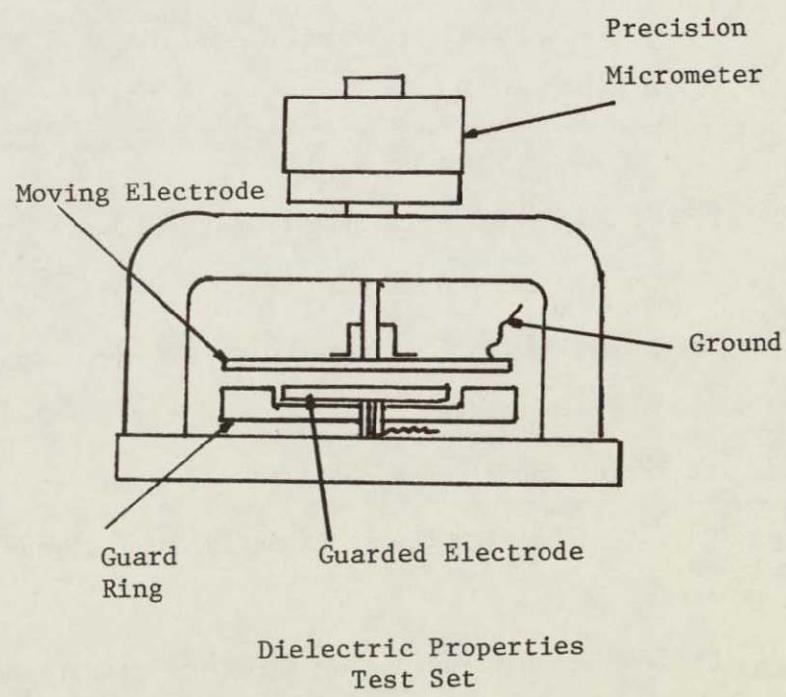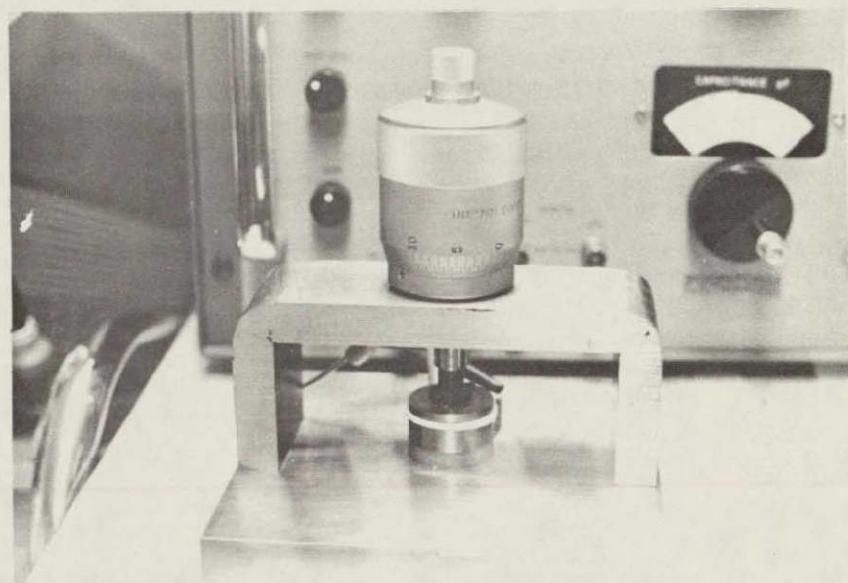
Figure 8.6

By noting the conductance on the bridge, the power factor may be found by:

$$p.f. = \frac{2G}{AF} \frac{t_2}{t_s - t_x} \times 9 \times 10^{11}$$

where   G = conductance in mhos

A = area of specimen in cm

f = frequency in Hz.

If the apparatus is connected to a Q-meter, the loss tangent may be calculated as follows.

Step 1) R_a, the equivalent parallel resistance of the apparatus, is calculated by:

$R_a = Q_a/\omega C_s$.

Step 2) R_{as}, the equivalent parallel resistance of the sample in combination with the apparatus, is calculated by:

$R_{as} = Q_s/C_s$.

Step 3) R_s, the equivalent parallel resistance of the sample, is calculated by:

$R_s = (R_a R_{as})/(R_a - R_{as})$.

Step 4)   The loss tangent is then calculated by:

Loss tangent = $1/\omega C_s R_s$.

Q_a is the Q reading on the Q-meter with the apparatus minus the specimen connected.

C_s is the series capacitance noted on the Q-meter.

Q_s is the Q reading with the specimen inserted in the apparatus.

ω is the frequency in radians at which the measurements are being made.

Table 8.38

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Ablebond A806-6

Dielectric Properties

Frequency (10^6 Hz)	Dielectric Constant		Loss Tangent	
	'Before Aging	After Aging	Before Aging	After Aging
1	5.16	5.71	.0074	.0069
5	5.46	5.71	.0072	.0077
10	5.37	5.19	.0067	.0070
20	5.32	5.26	.0073	.0087
30	5.33	5.13	.0079	.0159
40	5.25	5.06	.0071	.0107

Table 8.39

Epo-Tec H55

Dielectric Properties

Frequency (10^6 Hz)	Dielectric Constant		Loss Tangent	
	Before Aging	After Aging	Before Aging	After Aging
1	9.36	10.00	.0064	.0067
5	10.19	10.00	.0056	.0081
10	10.19	10.12	.0077	.0074
20	10.26	10.12	.0087	.0188
30	10.06	10.12	.0072	.0112
40	10.00	9.75	.0058	.0113

Table 8.40

Epo-Tec H61

Dielectric Properties

Frequency (10^6 Hz)	Dielectric Constant		Loss Tangent	
	Before Aging	After Aging	Before Aging	After Aging
1	8.68	7.83	.0053	.0068
5	8.68	7.83	.0047	.0068
10	8.59	7.83	.0065	.0085
20	8.25	7.83	.0087	.0186
30	8.42	7.55	.0079	.0109
40	8.34	7.91	.0064	.0131

Table 8.41

Epo-Tec H74

Dielectric Properties

Frequency (10^6 Hz)	Dielectric Constant		Loss Tangent	
	Before Aging	After Aging	Before Aging	After Aging
1	6.66	7.75	.0052	.0064
5	7.86	7.75	.0048	.0081
10	7.79	7.75	.0056	.0086
20	7.82	7.75	.0054	.0188
30	7.79	7.75	.0051	.0111
40	7.79	7.68	.0048	.0113

Table 8.42

Epo-Tec 921F1

Dielectric Properties

Frequency (10^6 Hz)	Dielectric Constant		Loss Tangent	
	Before Aging	After Aging	Before Aging	After Aging
1	8.34	9.74	.0071	.0075
5	9.38	9.76	.0062	.0067
10	9.22	9.65	.0079	.0083
20	9.32	9.43	.0072	.0182
30	8.87	9.33	.0074	.0111
40	9.02	9.22	.0066	.0150

Footnotes

[1] J.E. Zimmerman, "Measurement of Resistivity of Bulk Materials," <u>The Review of Scientific Instruments</u>, April, 1961, pp. 402-405.

[2] A.C. Lynch, "Measurement of Dielectric Properties of Low Loss Materials," <u>Proceedings of the Iee</u>, V112, Feb., 1965, pp. 426-431.

[3] Ibid.

Chapter IX

Characteristics of Various Epoxies

"Whenever epoxy adhesives are mentioned in a group of electronic professionals, it usually results in a division of the group into three camps. There are those who use epoxy with confidence, those who know little about how or when to use it, and thirdly, those who are reasonably sure it should not be used at all."[1] The first group has found that they can expect consistant results with adhesives, therefore they use them with confidence and ease. The second group, not really knowing anything about adhesives and not wanting to take any chances, use them only to repair or solve complex assemblies that cannot be solved by any other means. The third group have their own undisclosed reasons for not wanting to use epoxies. Do their reasons stem from reports of failures with epoxies or are their reasons based on some technical argument that the group has not yet shared with others in the industry? This chapter is designed to investigate the characteristics of various epoxies. The properties of five conductive epoxies, three silver filled and two gold filled, and two nonconductive epoxies will be examined. All these epoxies have been chosen at random.

Before looking at any specific adhesives, certain requirements must be set forth as a guideline to suitability for their use in micro-electronics. These properties are as follows:

    1. Adhesion: Epoxies must adhere to a variety of materials

                     including substrates, circuit components and

be able to retain their adhesive properties
during thermal exposure.

2. Thermal Stability: Epoxies must be able to withstand
elevated temperatures for long periods of
time without degradation in their mechanical
or electrical properties.

3. Chemical Stability: Epoxies must not cause corrosion on
any metallization. Outgassing must be such
as not to affect operation of the circuit.

4. Electrical Stability: Epoxies must maintain adequate
properties at and after prolonged exposure
to elevated temperatures.

5. Curing Temperature: Epoxies must be able to cure at
reasonable temperatures.

6. Pot and Shelf Life: Epoxies must be able to maintain
viscosities for a reasonable length of time.

7. Versatility: Epoxies must be able to be applied either
by hand, by automatic dispensers, or by
screen printing.

Table 9.1 gives some electrical characteristics expected of different
types of epoxies.[2]

The first epoxy to be considered is a one component, electrically
conductive epoxy developed by Ablestik Laboratories.[3] Ablebond 36-2
is comprised of a glycidyl ether resin, an aromatic substituted urea
and a filler of pure silver powder.[4] Ablebond 36-2 is a soft, smooth
paste designed for chip bonding in microelectronics. The smooth con-
sistency makes it ideal for dispensing by hand, by machine or by

Table 9.1

Adhesive Requirements for Hybrid Assembly

Adhesive Type	Usage	Physical Properties
Electrically Conductive		
A. Paste	Chip and Substrate attachment and for grounding	Volume resistivity less than 100 $\mu\Omega$-cm
B. Paste, Moisture resistant	Lid seal	Fair volume resistivity, ability to form hermetic seal
C. Preform, Film	Lid seal	Excellent moisture resistance
Thermally Conductive	Component attachment	Thermal conductivity less than 2 x 10^{-2} watts/cm°C
Insulating Pastes		Common to all $\rho = 10^{11}$ $\mu\Omega$-cm
A. Shape retaining	Component attachment	Maintain shape through cure
B. Moisture Resistant	Lid seal repair Component attachment	Excellect moisture resistance
C. High Temperature Resistant	Component attachment	Maintain good bond after short exposures to 300°C
D. Fast cure at 25°C	Component attachment	Cure in 15 to 30 seconds at 25°C. Low viscosity

screen printing. Ablebond 36-2 has a shelf life of about six months if stored at -40°C and a pot life of one week at room temperature. The bond strength is greater than 2.4 x 10^7 N/m^2 at room temperature with little or no degradation after aging for 1000 hours at 150°C. The bond strength decreases slightly during temperature cycling.[5] This solvent free epoxy causes very little outgassing after cure. Outgassing increases only slightly due to aging (Fig. 9.1).[6] Ablebond 36-2 has an average resistivity of 1.6 x 10^{-4} ohm-cm at room temperature (Fig. 9.2) with a cold TCR (-55° to)°C) of 2525 ppm/°C, and a hot TCR (0° to 150°C) of 2331 ppm/°C. The resistivity of Ablebond 36-2 decreased by a slight three per cent during an aging period of 1000 hours at 150°C (Fig. 9.3).[7] Ablebond 36-2 (either cured or uncured) shows little or no corrosive effects on any metalization.[8]

The second epoxy, also from Ablestik Laboratories, is a gold filled conductive epoxy designed for chip bonding. Ablebond 58-1 is a bisphenol based epoxy cured with an aromatic substituted urea.[9] This epoxy is also a smooth paste designed for dispensing either by hand, by machine, or by screen printing. The shelf life for Ablebond 58-1 is approximately six months at -40°C and the pot life is about two weeks at room temperature. Curing time is recommended as one-half hour at 150°C. Bond strengths of this epoxy range from 9.3 x 10^6 N/m^2 at 150°C to more than 2.4 x 10^7 N/m^2 at room temperature. Slight degradation in strength has been observed after prolonged exposure to high temperatrures.[10] Although a higher outgasser than Ablebond 36-2, this epoxy tends to level off much faster as shown in Figure 9.4.[11] Ablebond 58-1 has been found to have little if any corrosive effects on any metallization.[12] Results of pyrograms show that Ablebond 58-1

Aging Time (Days)
Ablebond 36-2

Figure 9.1
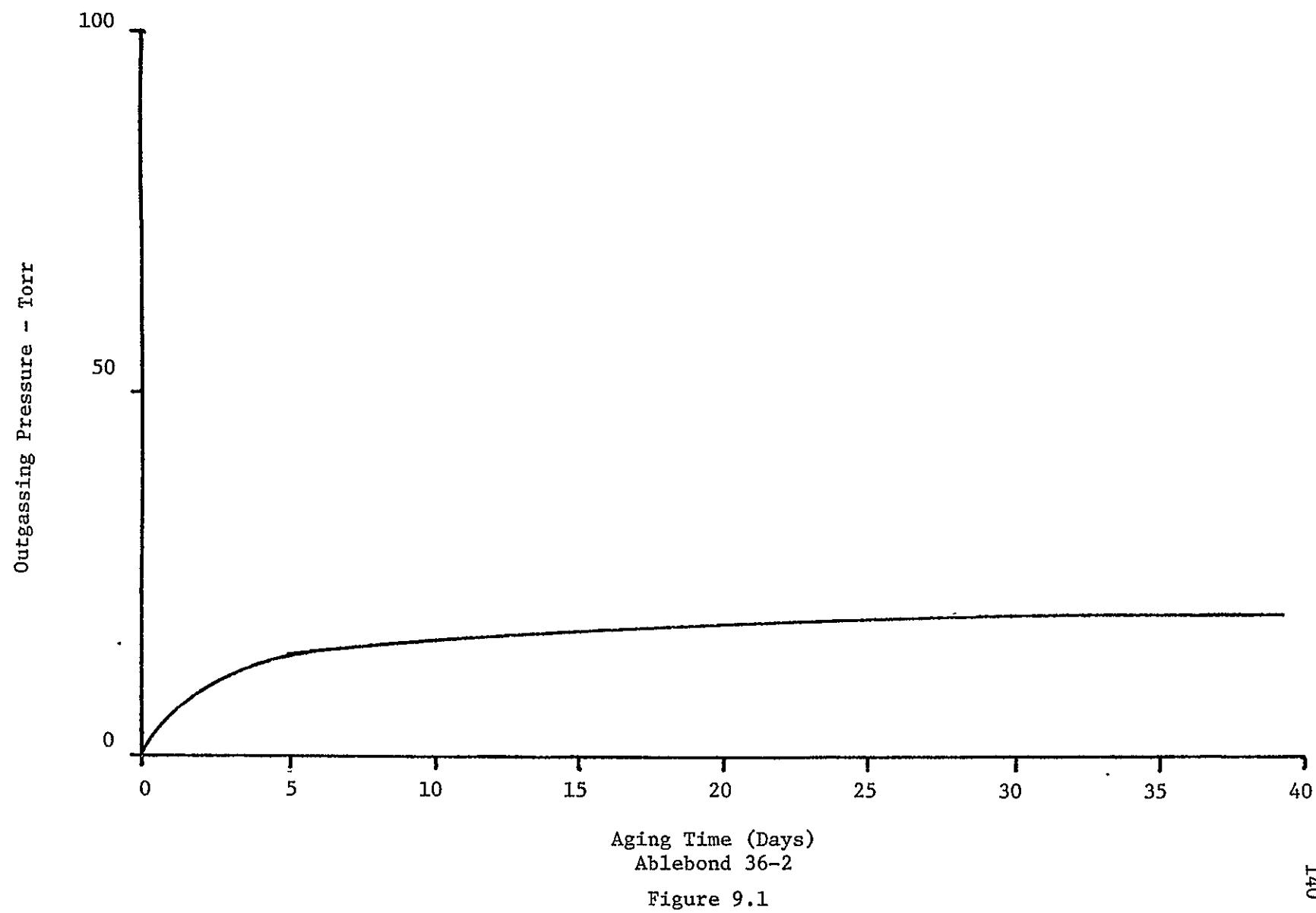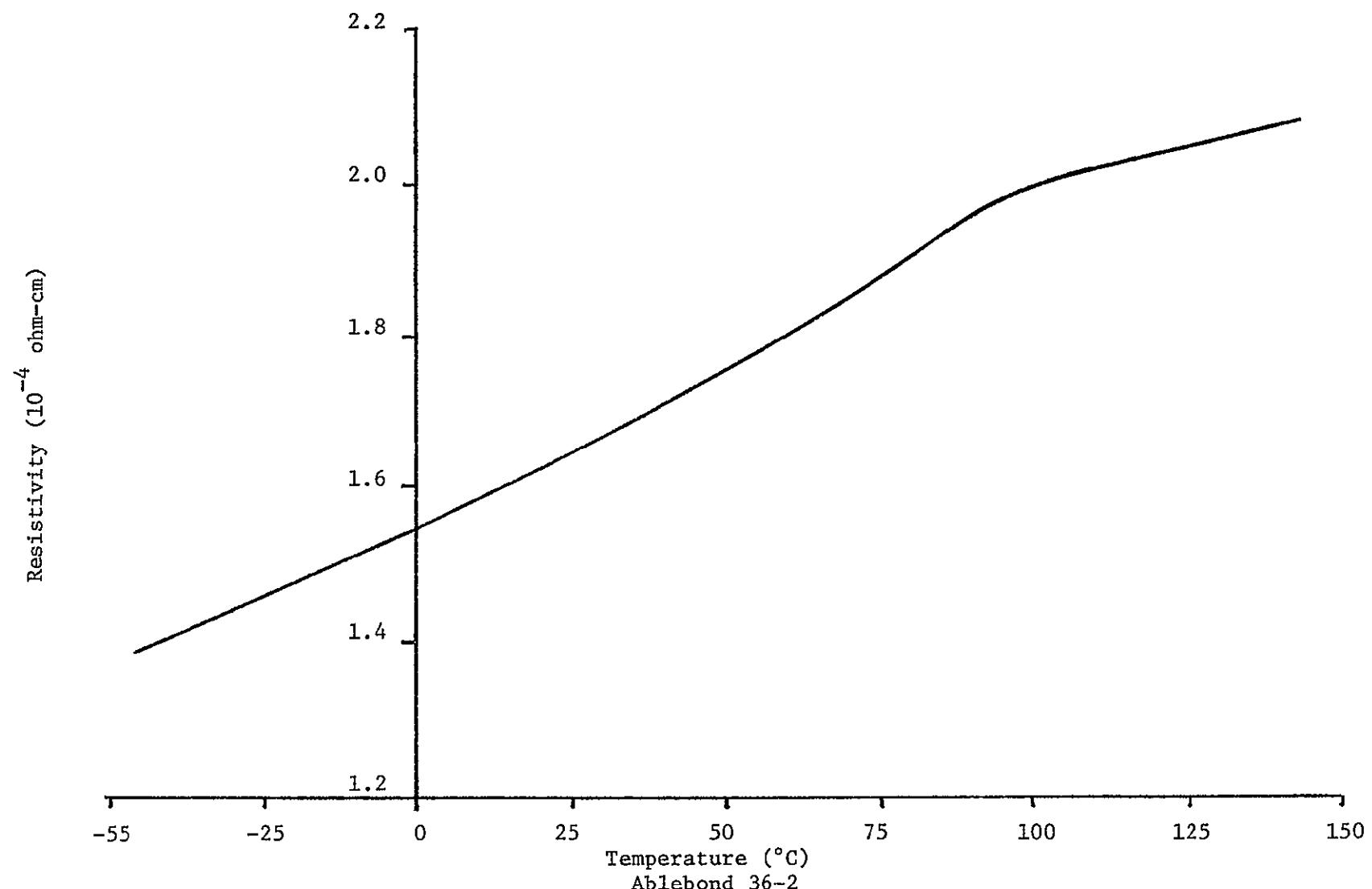
Ablebond 36-2

Figure 9.2

Figure 9.3

Aging Time – Days at 150°C
Outgassing of Ablebond 36-2 after cure
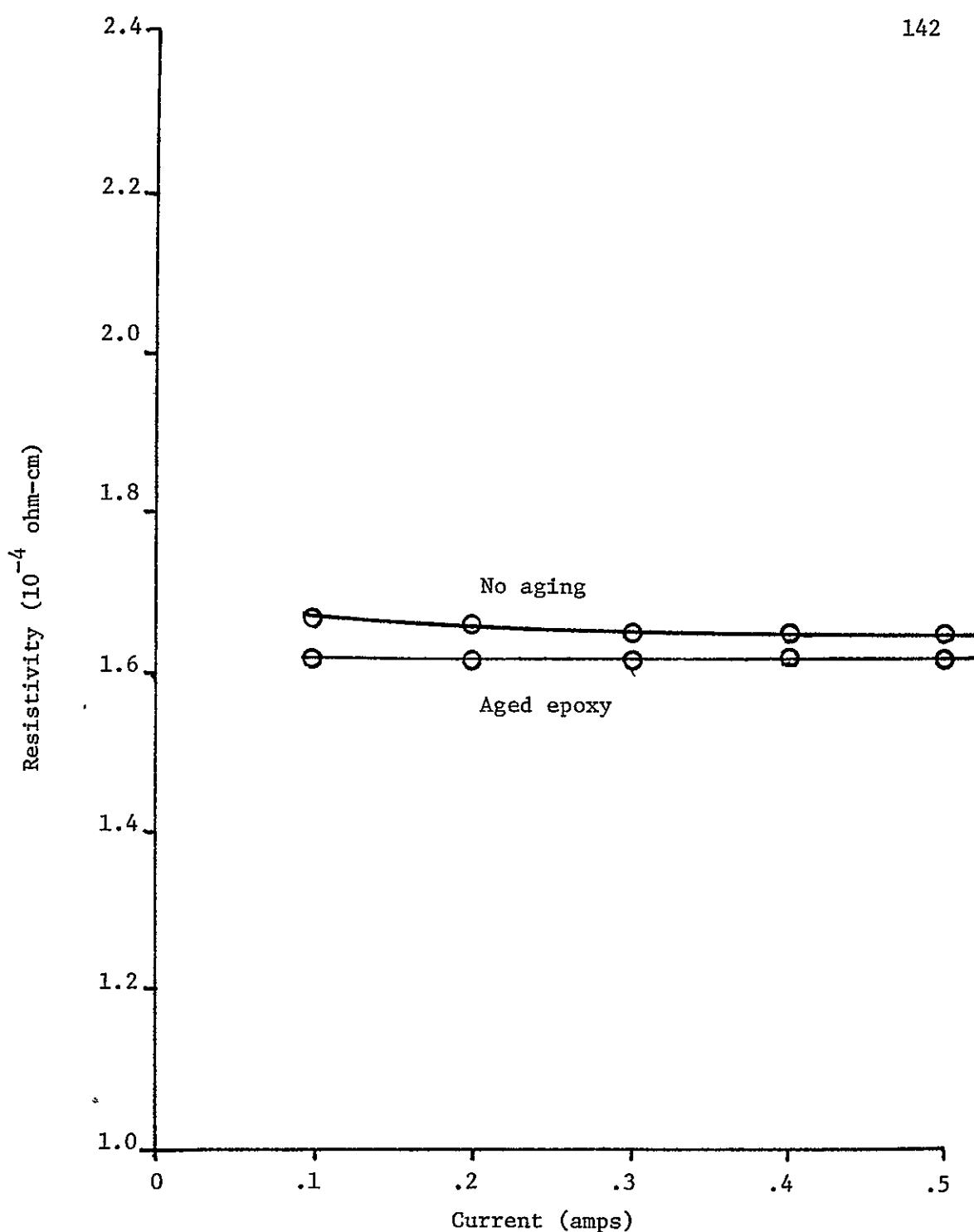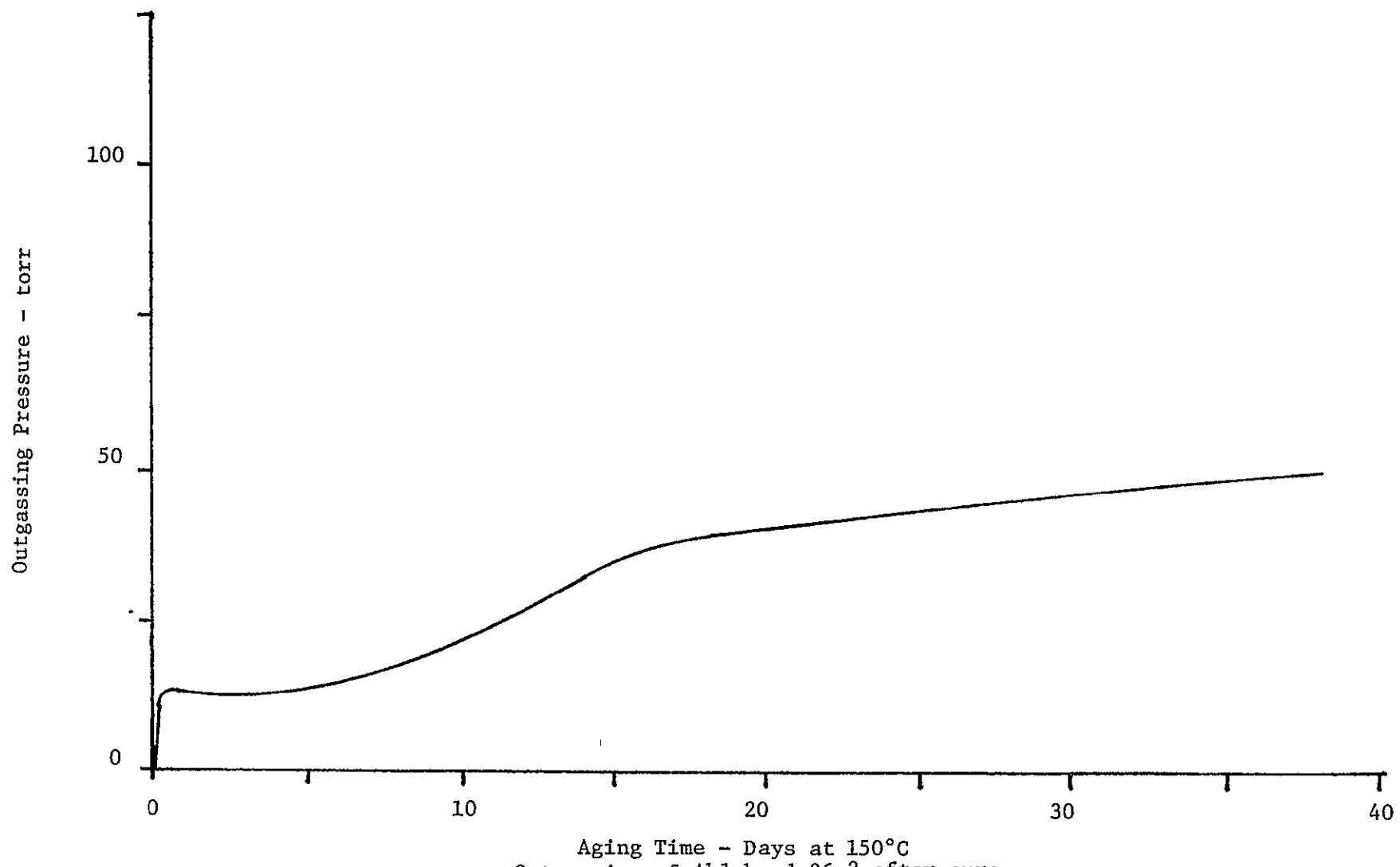
Figure 9.4

has a lower weight loss than Ablebond 36-2 (Fig. 9.5).[13] The electrical

resistivity at room temperature is about 6.26×10^{-4} ohm-cm which is

about two orders of magnitude greater than that of pure gold (Fig. 9.6).

A decrease of three per cent can be seen after aging at 150°C for 1000

hours. Ablebond 58-1 has a cold TCR of 1470 ppm/°C and a hot TCR

of 3800 ppm/°C.[14]

Another silver filled conductive epoxy with much the same

properties of the Ablebond epoxies is offered by Epoxy Technology.

Epo-Tec H-31 is a solvent free epoxy designed for chip bonding of

resistors, transistors, capacitors, etc. Epo-Tec H-31 was designed to

operate at temperatures from 300° to 350°C for wire bonding purposes.

Curing temperatures range from 100°C for one hour to 150°C for one-

half hour. The shelf life is approximately six months when stored at

room temperature. Bond strengths of this epoxy range from 1.6×10^7 N/m^2

at 150°C to 2.3×10^7 N/m^2 at room temperature and show little de-

gradation due to aging.[15] This epoxy outgasses substantially as seen

in Figure 9.7, but is essentially coincident with Ablebond 36-2 in

weight loss to about 400°C (Fig. 9.8).[16] Epo-Tec H-31 has an average

resistivity of 4.07×10^{-4} ohm-cm (Fig. 9.9) at room temperature with

a cold TCR of 4124 ppm/°C and a hot TCR of 2326 ppm/°C. A four per

cent change in resistivity occurred during aging (Fig. 9.10).[17]

Epo-Tec H-31 has been found to be corrosive to thin film aluminum such

as might be used in hybrid microelectronics. Corrosion was also evi-

dent on gold film metallization. NASA, at this time, is conducting

experiments to determine the exact effects of corrosion and the causes.[18]

One possible explanation is the use of boron-trifluoride as a curing

agent. This catalyst was seen in Chapter II to be highly corrosive.
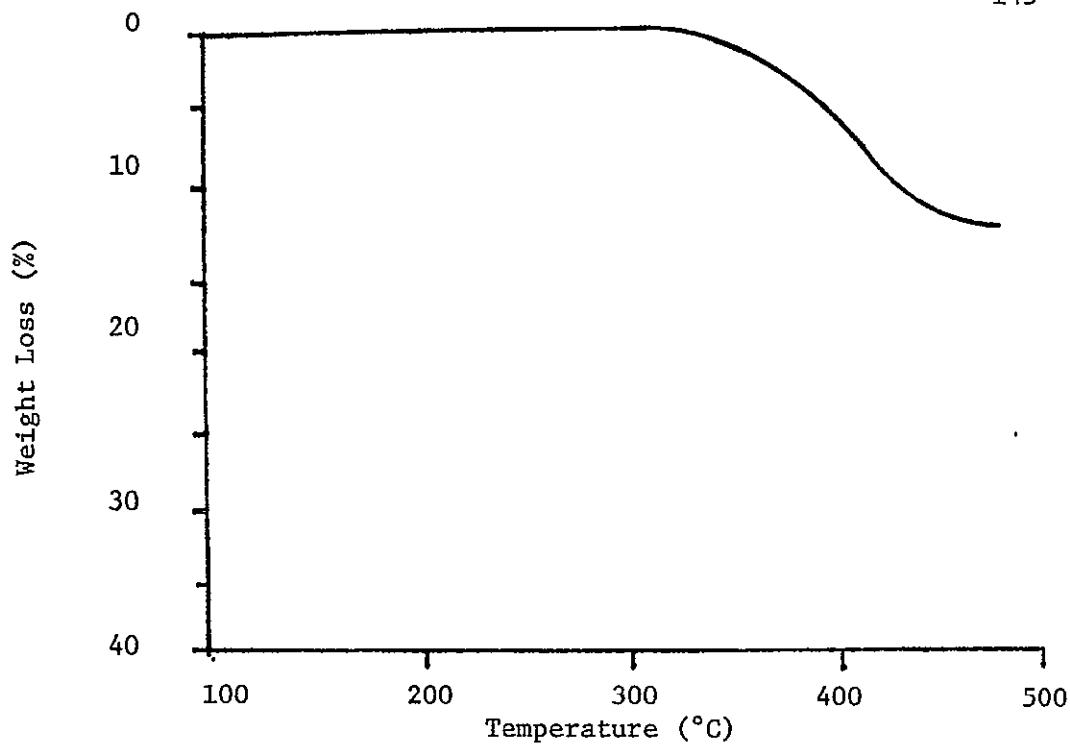
TGA curve – Ablebond 58-1
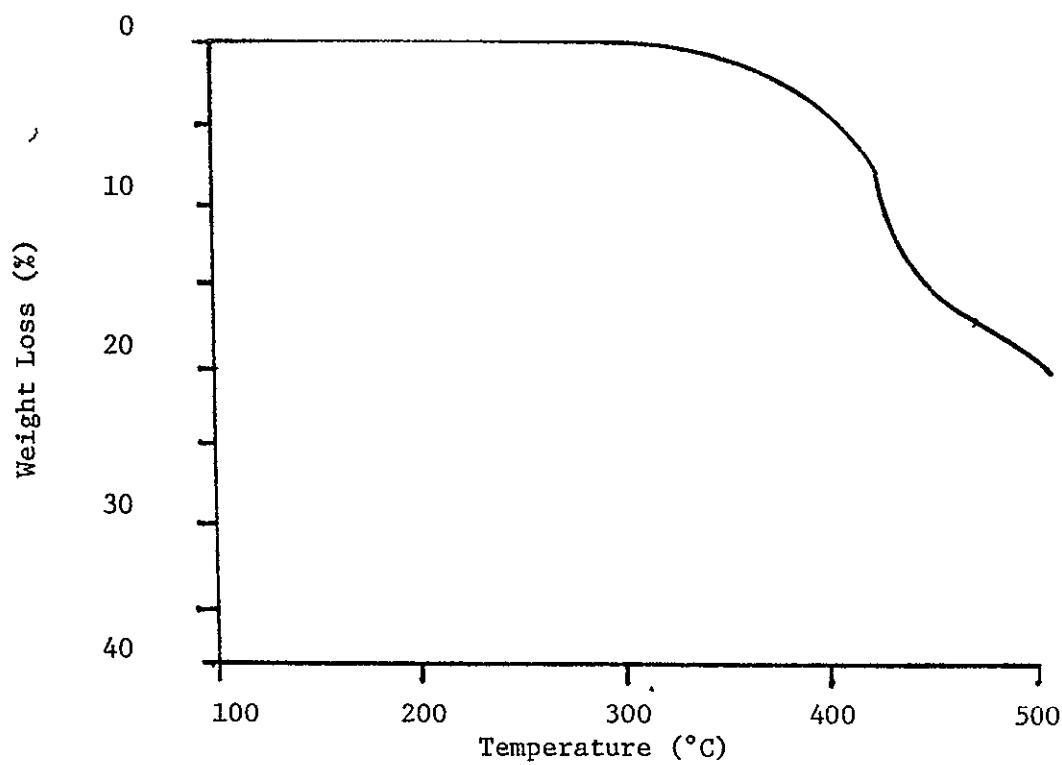
TGA curve – Ablebond 36-2

Figure 9.5

Figure 9.6

Ablebond 58-1

Outgassing Pressure - torr

100

50

0

0　　　　　　　　　　10　　　　　　　　　20　　　　　　　　　30

Aging Time — Days
Outgassing of Epo-Tec H31

Figure 9.7

TGA curve – Epo-Tec H31
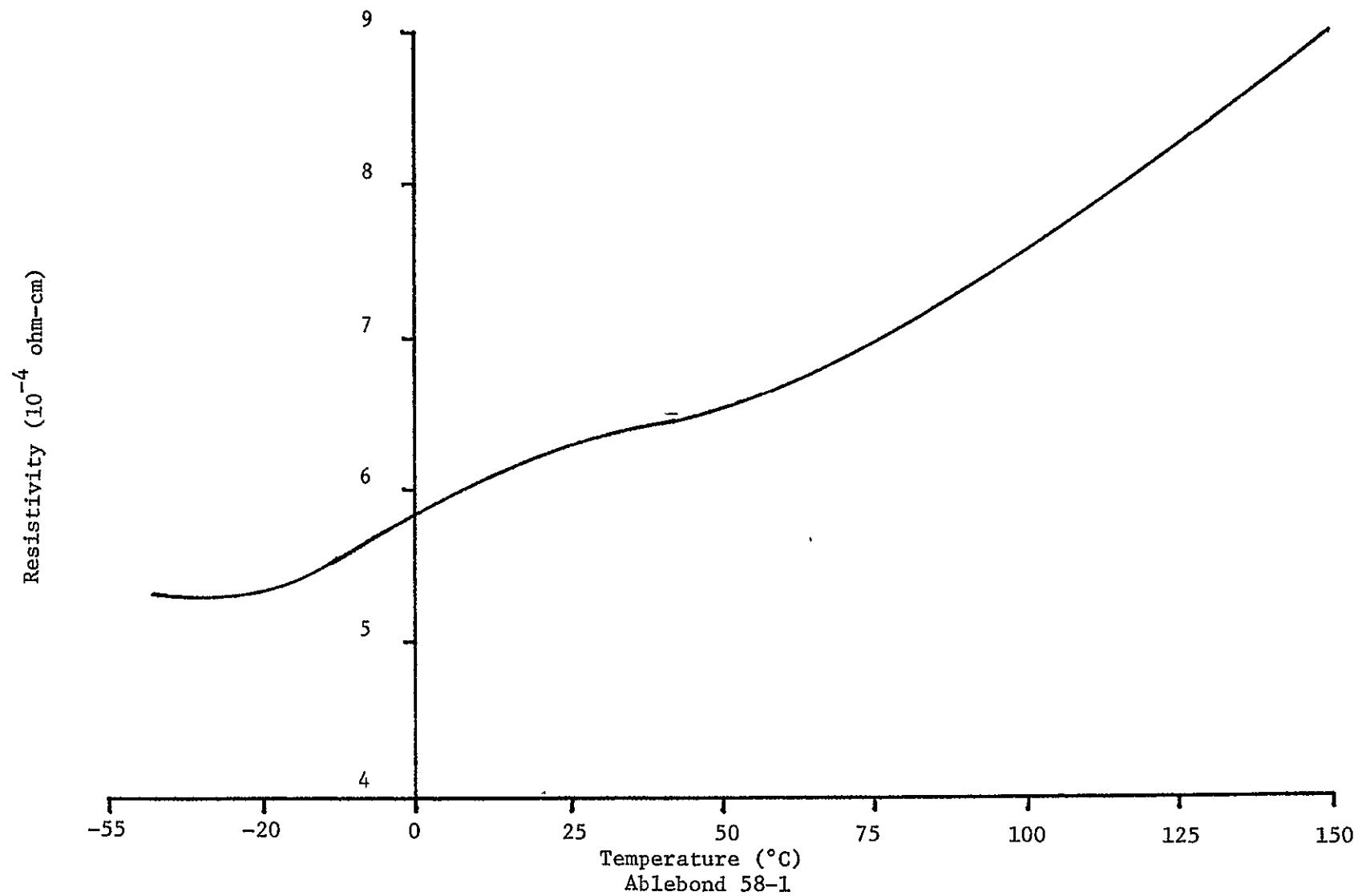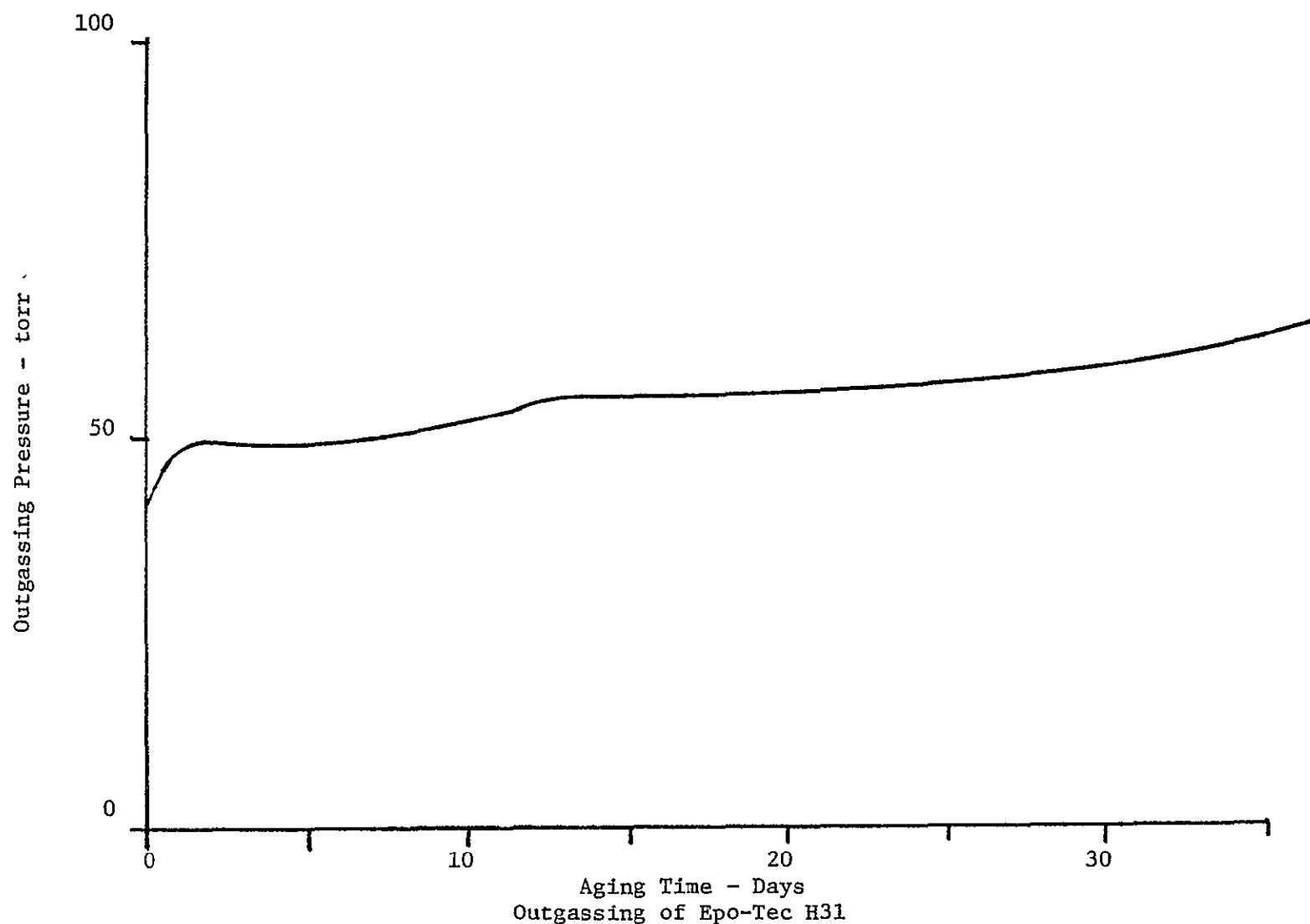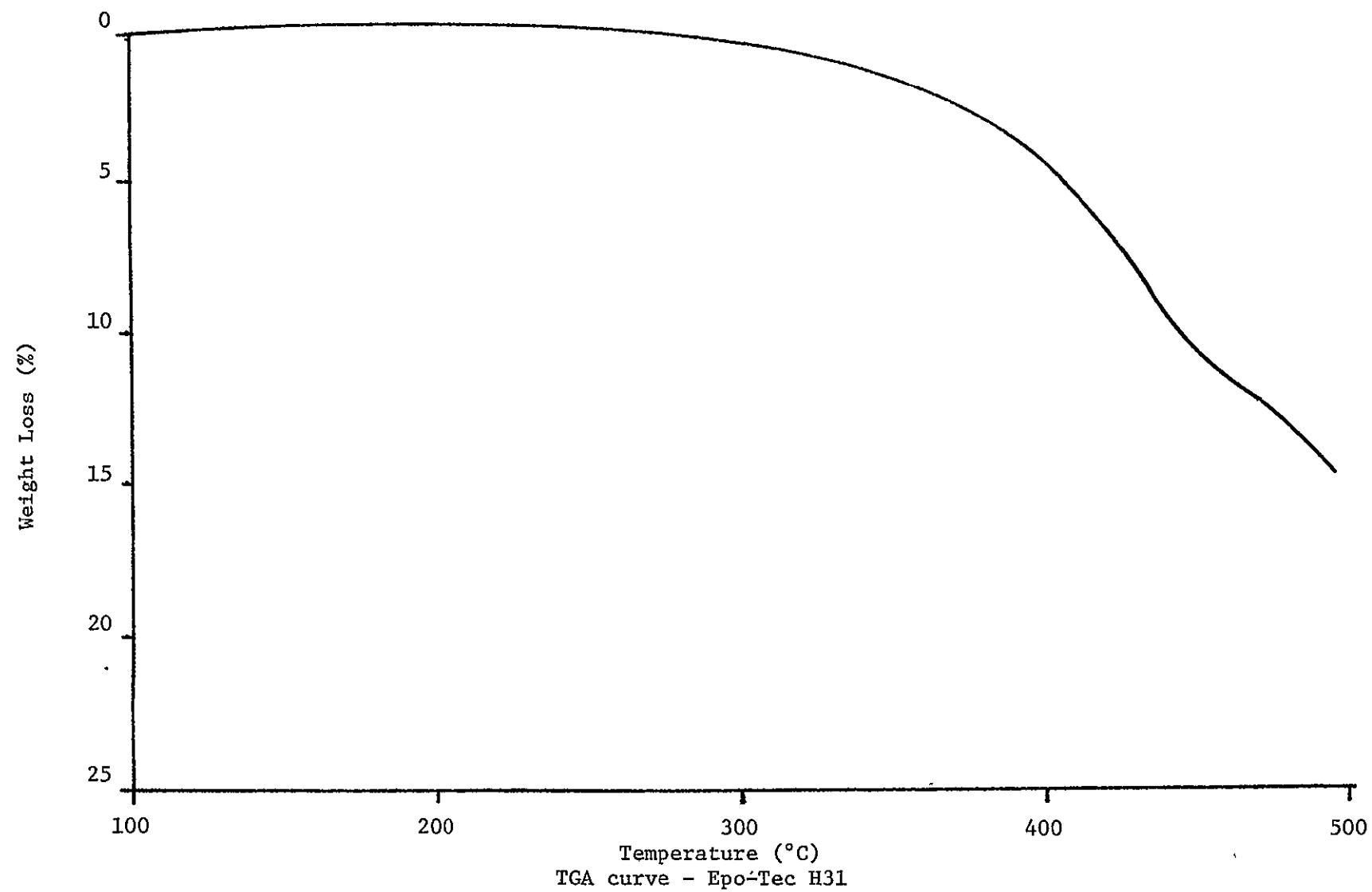
Figure 9.8
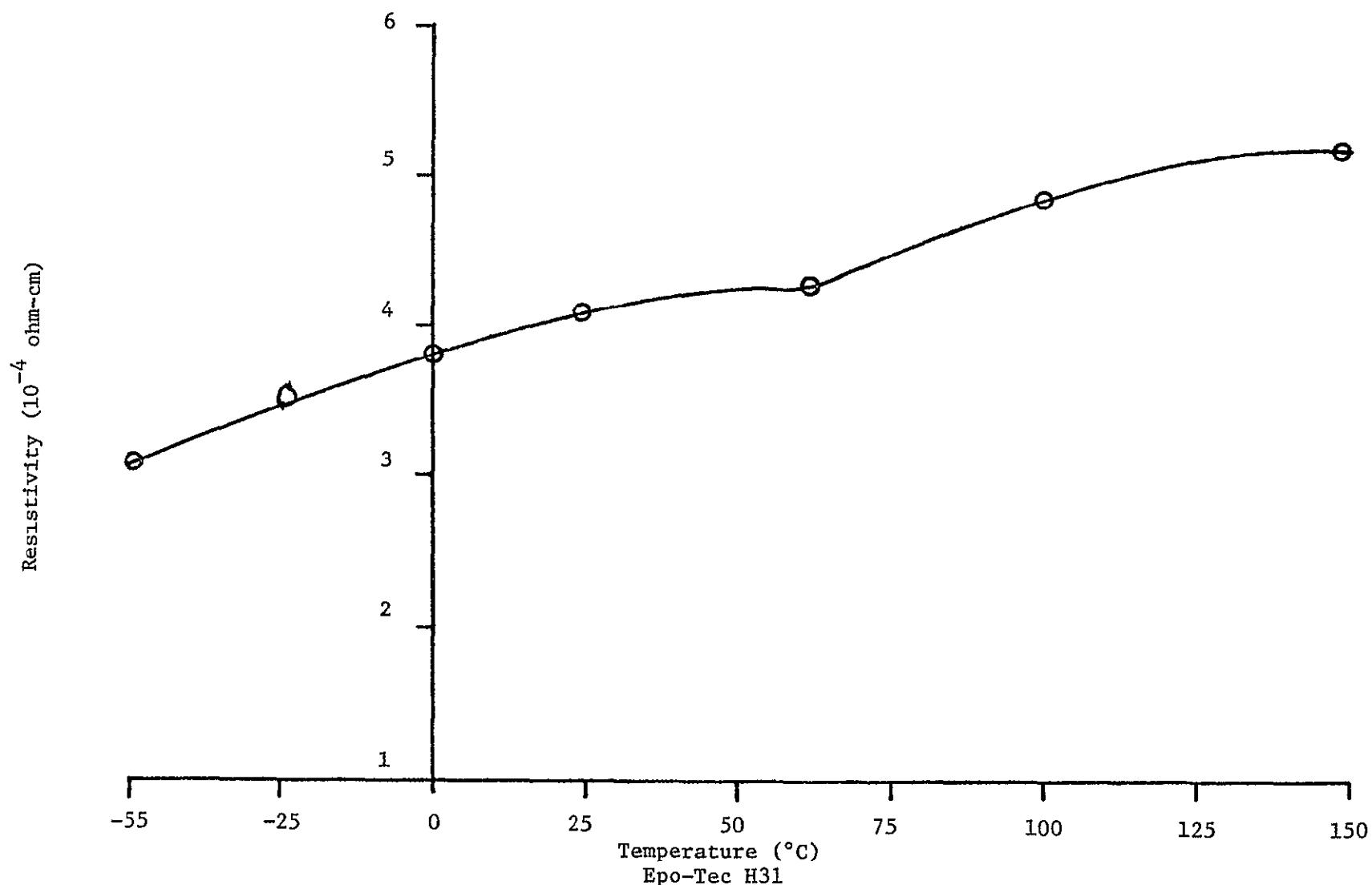
Figure 9.9

Epo-Tec H31

Epoxy Technology also offers a gold filled conductive epoxy designed especially for chip bonding. Epo-Tec H-44, an amine cured epoxy, contains no solvents thus "drying out" does not present a problem. Its soft smooth paste allows for any manner of application and its long shelf life permits application days before curing is to take place. H-44, like H-31 is designed to operate at temperatures from room temperature to over 300°C. The recommended curing time is one hour at 100°C. H-44 has a bond strength of about 2.43×10^7 N/m^2 at most temperatures. Like the other epoxies discussed, H-44 remains unaffected or only slightly degraded in bond strength by prolonged aging.[19] The considerable outgassing found with H-44 could be indicative of decomposition within the epoxy (Fig. 9.11).[20] Corrosion was evident on thin film aluminum when exposed to H-44, however, gold metallization showed no effects.[21] The electrical resistivity of H-44 was somewhat higher than that of the silver filled epoxies--the average being about 8.58×10^{-4} ohm-cm at room temperature (Fig. 9.12). Resistivity was found to decrease by three percent after aging. The cold TCR of H-44 is about 2165 ppm/°C with the hot TCR being about 2879 ppm/°C.[22]

Two component epoxies are also available in gold and silver. Epoxy Technology offers a silver filled, two component, conductive epoxy, H-20E. Part A contains the resin and a silver powder; part B contains the hardener and a silver powder. This particular epoxy can be mixed either by weight or by volume. The manufacturer suggests a one to one ratio of resin to hardener by weight or by volume. Curing temperatures are as low as 50°C. H-20E has an exceptionally long shelf life, two years, when stored at room temperature, but the pot life of

Figure 9.10

Outgassing of Epo-Tec H44
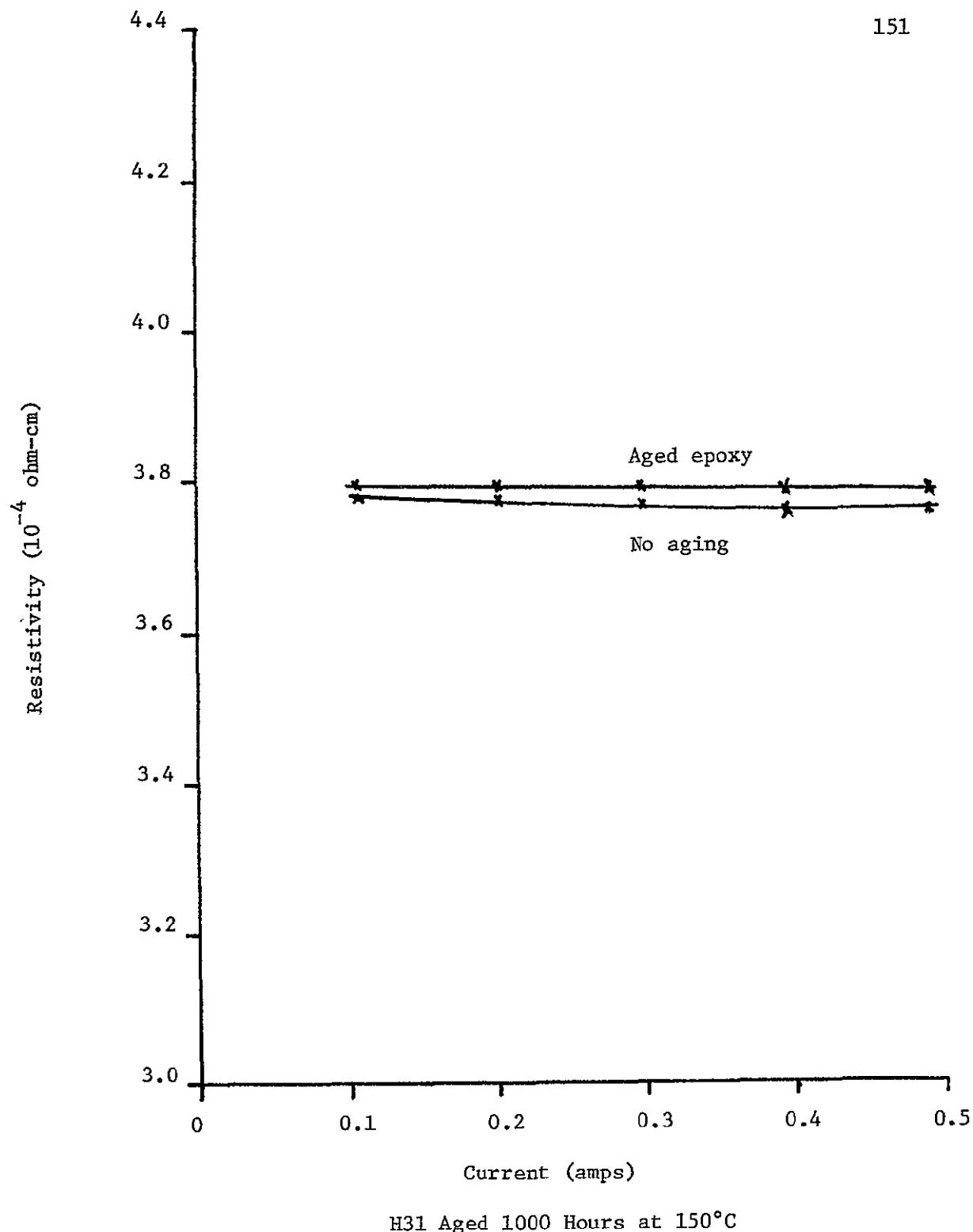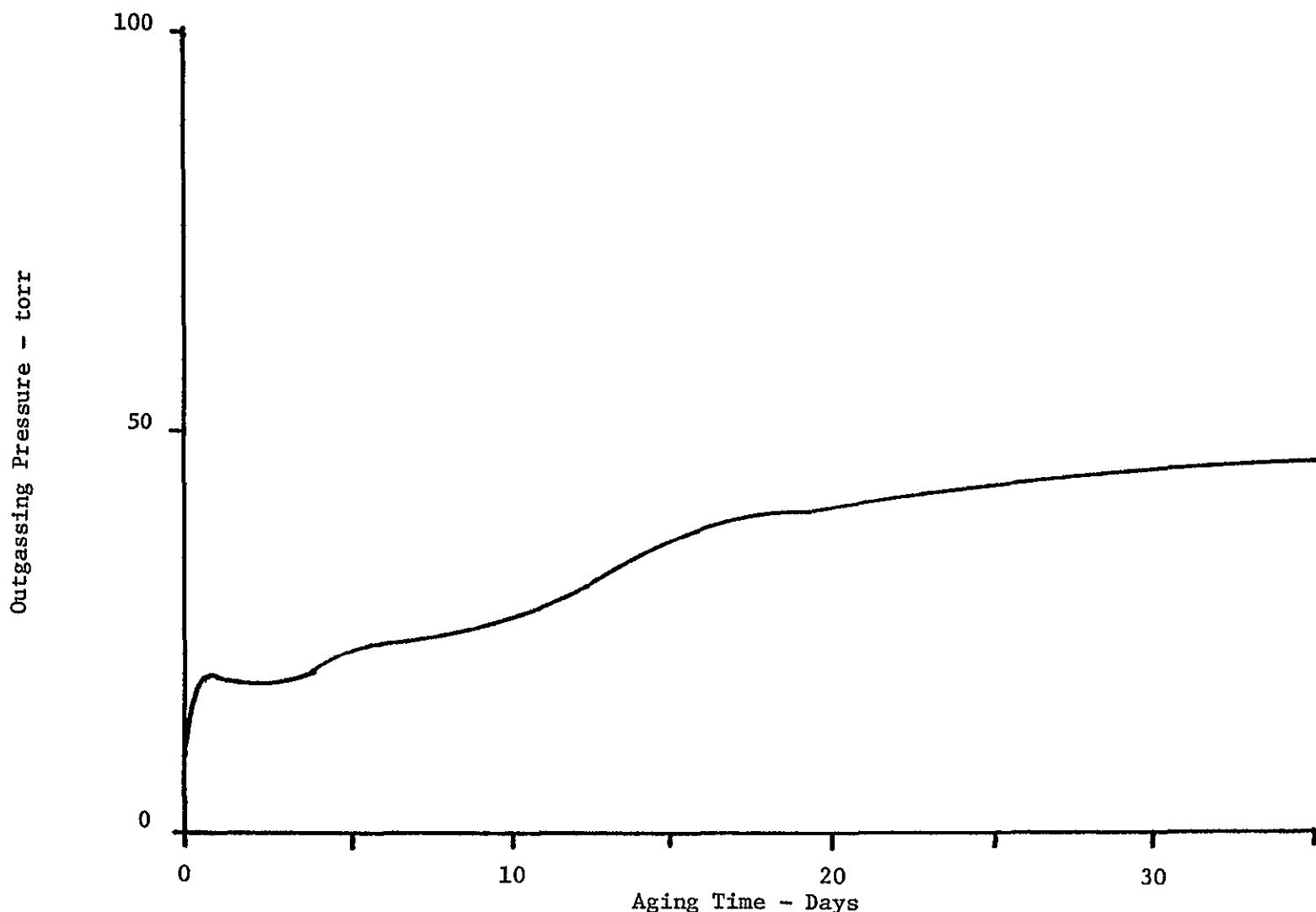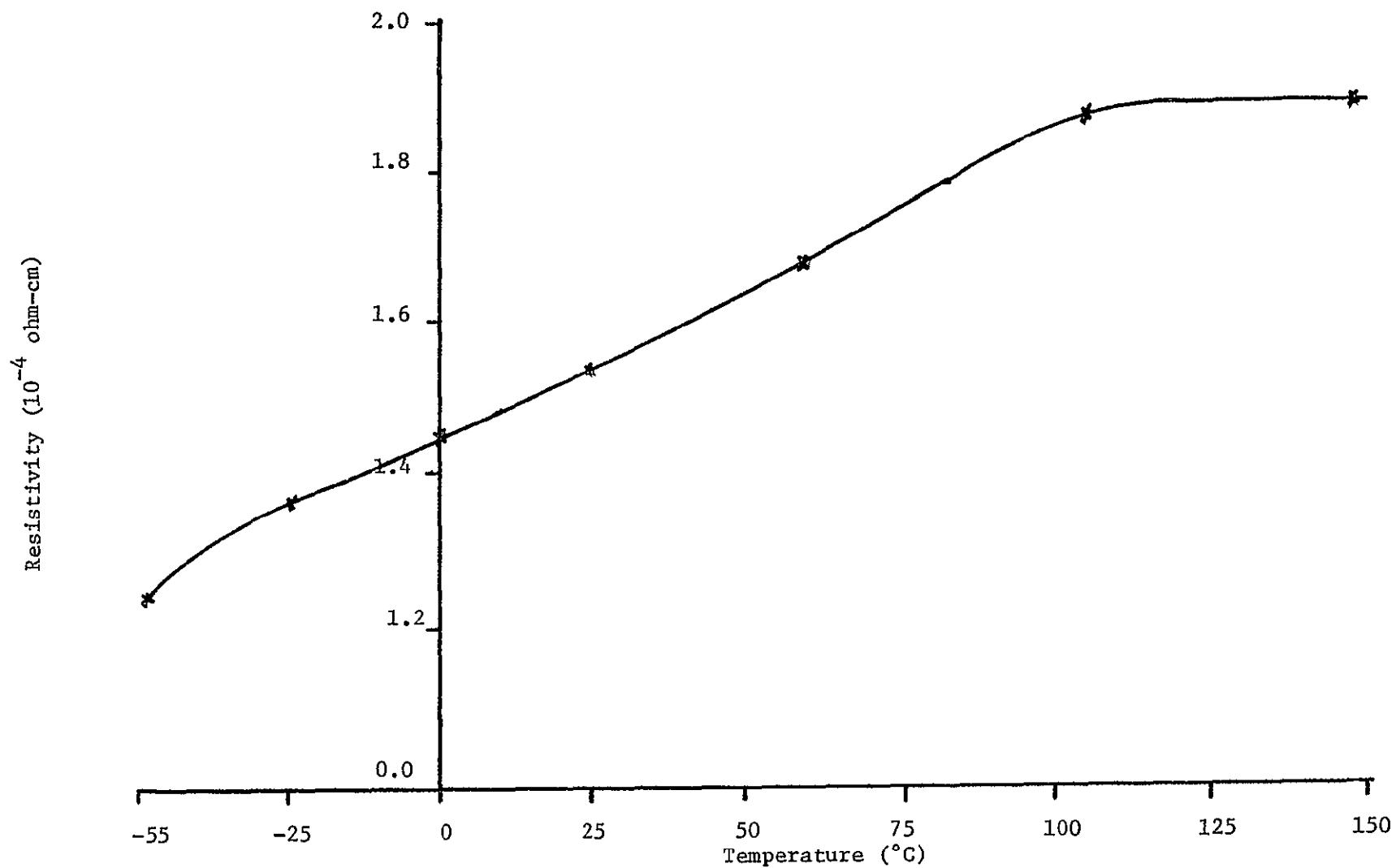
Figure 9.11

Epo-Tec H20E

Figure 9.13

four days requires that the epoxy be used soon after mixing. When mixed H-20E is also a soft smooth thixotropic paste designed for any method of application. The bond strength of H-20E is in the neighborhood of 1500psi at room temperature and, like the others, is only slightly reduced by aging. The average resistivity at room temperature is seen to be 1.55×10^{-4} ohm-cm (Fig. 9.13) which varies only slightly with temperature. Aging shows a slight three per cent decrease in volume resistivity.[23]

Electrically insulative epoxies also play a very important part in microelectronics. Applications of these epoxies include bonding chips to substrates where extra strength is needed or to hold the chips in place in order to wire bond. Nonconductive, or insulative, epoxies can also be used as a protective covering or as a lid sealant. These epoxies, like conductive epoxies, also come in one- or two-component forms. They can be either thermally conductive or thermally insulating, but either way they are generally good electrical insulators.

Epoxy Technology offers a two-component electrically insulative epoxy, H-55. This amine cured novolac epoxy is also thermally insulating. The mixing ratio for this epoxy is recommended as twenty parts (by weight) of part A (resin) to one part of part B (hardener). H-55 obtains its final bond properties after curing at 100°C for twenty minutes. Shelf life is estimated to be one year at room temperature with a pot life of about four days. Bond strengths are rated at 7.2×10^{6} N/m^2 at room temperature to 1.9×10^{6} N/m^2 at 150°C.[24] Volume resistivity is about 3×10^{15} ohm-cm, the loss tangent about .007, and the dielectric strength is about 400 V/mil. The dielectric constant rates about 10 as compared to air which is normally 1 (Table 9.2).[25]
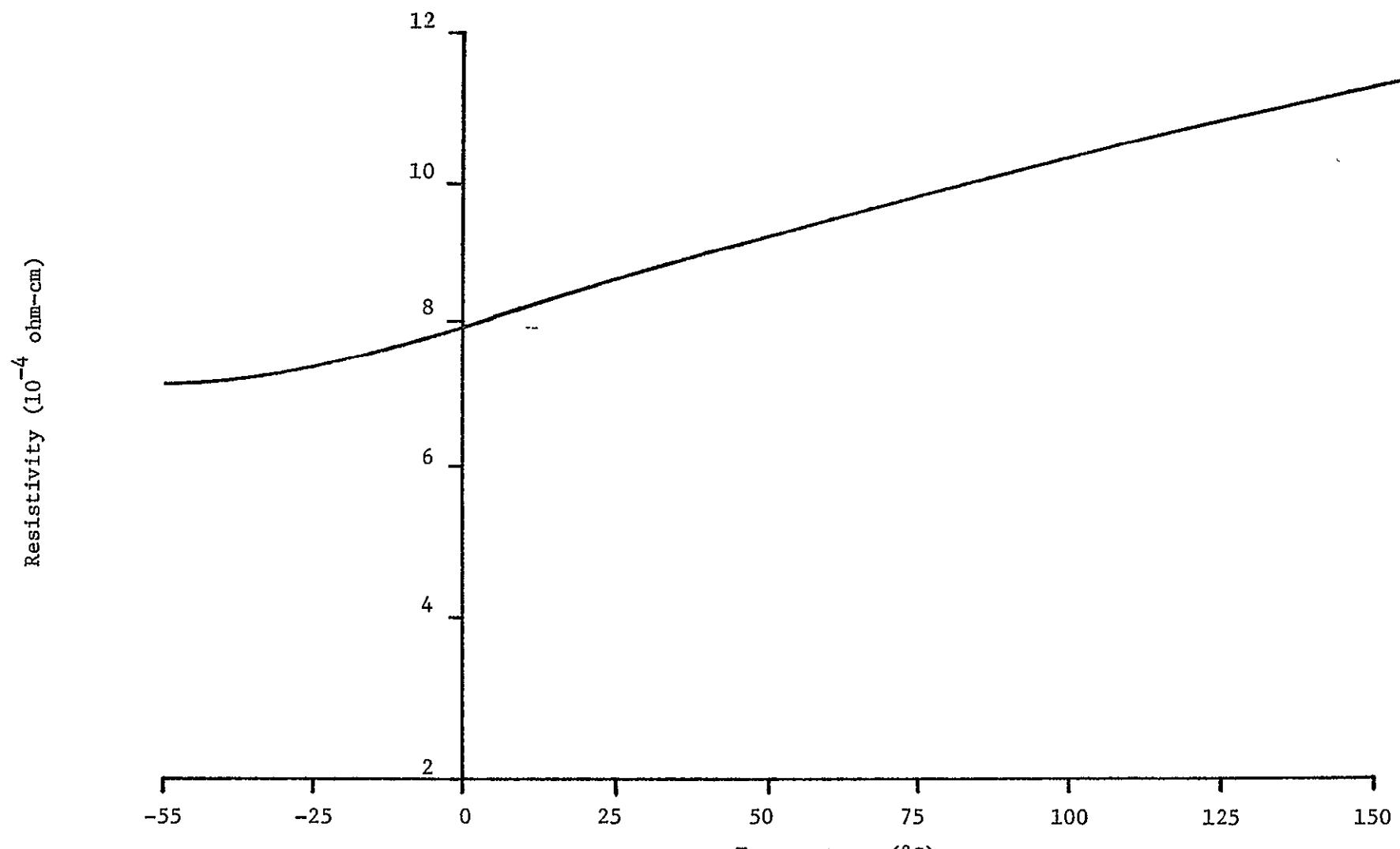
Epo-Tec H44

Figure 9.12

Table 9.2

Epo-Tec H-55

Dielectric Constant

Frequency (Hz)	Dielectric Constant
1×10^6	9.357
5×10^6	10.190
10×10^6	10.190
20×10^6	10.260
30×10^6	10.060
40×10^6	10.000

Ablestik Laboratories offers an insulating epoxy, A806-6, based on bisphenol and "Hydantoin" resins, cured with an aromatic subst-tuted urea, and filled with silica.[26] This one-component epoxy has a pot life of about one week at room temperature bu will remain usable for about six months if stored in a refrigerator. Ablebond A806-6 has bond strengths from 500 psi to 1900 psi. Volume resistivity approaches 4.5×10^{14} ohm-cm. The dielectric constant is about 5.4 with a loss tangent of .007 (Table 9.3).[27]

As can be seen, epoxies are available in all different types. They can be obtained in either one- or two-part forms or in either conductive or nonconductive forms. In the conductive forms, the epoxies come in either gold filled or silver filled and in some cases even copper filled (not readily available). Their consistency makes them very easy to use and their curing temperatures are low enough to prevent component damage but yet cure thoroughly. With proper usage, epoxies can be a great help to the hybrid manufacturer.

Table 9.3

Ablestik A806-6

Dielectric Constant

Frequency (Hz)	Dielectric Constant
1×10^6	5.161
5×10^6	5.461
10×10^6	5.396
20×10^6	5.316
30×10^6	5.333
40×10^6	5.246

Footnotes

[1]D.D. Zimmerman, "Electrical Use of Epoxy Adhesives in Microelectronics." (Author is source)

[2]Peter J. Planting, "An Approach for Evaluating Epoxy Adhesives for Use in Hybrid Microelectronic Assembly," IEEE Transactions on Parts, Hybrids, and Packaging, Dec., 1975, pp. 305-311.

[3]All data has been taken from epoxy specification sheets given by by the manufacturer unless otherwise noted.

[4]Norm Nygard, sales representative, Ablestik Laboratories, private communication.

[5]Caruso, S.V., "Design Guidelines for Use of Adhesives and Organic Coating in Hybrid Microelectronics," NASA Technical Memorandum TM X-64908, Dec., 1974, p. 21.

[6]Ibid., p. 18.

[7]Cree W. Stout, J.E. Sergent, S.V. Caruso, "Electrical Properties of Adhesives Used in Hybrid Microelectronic Applications," Electronic Components Convention, San Francisco, April, 1976.

[8]Caruso, p. 33.

[9]Nygard.

[10]Caruso, p. 16.

[11]Ibid., p. 21.

[12]Ibid., p. 33.

[13]Ibid., p. 42.

[14]Stout, et al.

[15]Caruso, p. 51.

[16]Caruso, p. 51.

[17]Stout, et al.

[18]Caruso, p. 34.

[19]Ibid., p. 51

[20]Ibid., p. 21.

[21]Ibid., p. 33.

[22]Stout, et al.

[23]Ibid.

[24]Caruso, p. 51.

[25]Stout, et al.

[26]Nygard.

[27]Stout, et al.

Chapter X

Conclusions

Having examined some typical epoxies, the following conclusions
may be drawn.

1. One component epoxies are much simpler to use since
    they require no mixing.

2. One component epoxies are more economical. The unused
    portion may be refrigerated and used later. Two-
    component epoxies, however, have a longer shelf life.
    For small production runs, the one component epoxies
    may prove to be more economical.

3. Two component epoxies are more reliable than one
    component epoxies.

4. Two component epoxies, in general, have a lower curing
    temperature due to the amount of·inhibitor put into
    the one component epoxies to prolong shelf life. However,
    a longer curing time than specified by the manufacturer
    is strongly recommended.

Insulative epoxies are primarily used for chip bonding and
lid sealing. Some properties are as follows:

1. The epoxy cures at a low temperature which is necessary
    for lid bonding in that circuit components may not be
    able to withstand high temperatures.

2. When sealed with epoxy, the package is easily repaired.

3. Epoxy may be used on any material; whereas, welding and
    soldering is limited to metals.

All of the epoxies that have been studied are adaptable to any

method of dispensing. If dispensed by hand, the results are nonre-

producible as well as the technique being time consuming. Screen

printing is less time consuming and is, therefore, preferred over

hand dispensing. Screen printing produces a thin coat over all areas

at one time. This thin coat is especially desirable when working with

power devices. Machine dispensing, although expensive, is also a good

way of dispensing epoxy. This method can be adjusted so as to be able

to place as much epoxy as is desirable.

Mechanical strengths of epoxies, from all studies performed,

have proved to be excellent. However, as the ambient temperature

increases, the mechanical strengths of the epoxy decreases. If the

temperature is high enough and with some amount of stress, the epoxy

bond can fail causing circuit problems.

After having made considerable research into the chemical

makeup and other properties of epoxies, this author can only join

the group mentioned in Chapter IX in using epoxies with confidence

and ease. However, one must realize that there are different epoxies

for different applications and must weigh all the characteristics

of the epoxy against the specified requirements for use. With careful

matching of need and epoxy, and by following the manufacturer's

directions, there should be no cause for alarm when using epoxies.

Appendix

By assuming that:

1. The wave length is much greater than the dimensions of
the sample in question, then the displacement current may
be neglected.

2. A time-varying field is in order.

3. There is symmetry about the axis, i.e. $\overline{i_z}$ is a function
of the radius r.

and replacing the electric field given by Smythe[1] with a magnetic field,
the total flux can be obtained and hence the reactance and the resistance.

$$\nabla \times \overline{E} = -\frac{d\overline{B}}{dt} \qquad \text{Maxwell's equation} \qquad \text{(Eq. A1)}$$

$$\overline{B} = \nabla \times \overline{A}$$

$$\nabla \times \overline{E} = -\frac{d}{dt}(\nabla \times \overline{A})$$

$$\overline{E} = -\frac{d\overline{A}}{dt} \qquad \text{(Eq. A2)}$$

$$\overline{i} = \sigma\overline{E} \qquad \begin{array}{l} \text{Definition of} \\ \text{conductive current} \end{array}$$

$$\nabla \times \overline{i} = \nabla \times \sigma\overline{E}$$

$$= \sigma\nabla \times \overline{E}$$

$$= \sigma(-\frac{d\overline{B}}{dt})$$

$$\sigma = 1/\rho$$

$$\rho\nabla \times \overline{i} = -\frac{d\overline{B}}{dt} \qquad \text{(Eq. A3)}$$

$$= -\frac{d}{dt}(\nabla \times \overline{A})$$

$$= -\nabla \times \frac{d\overline{A}}{dt}$$

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$$\bar{i} = -\frac{d\bar{A}}{dt} \qquad \text{(Eq. A4)}$$

$$\bar{B} = \mu\bar{H}. \qquad\qquad \text{Definition of permeability}$$

$$\nabla \times \bar{H} = \bar{i} \qquad\qquad \text{Maxwell's equation}$$

$$\nabla \times \frac{\bar{B}}{\mu} = \bar{i}$$

$$\frac{1}{\mu} \nabla \times \bar{B} = \bar{i}$$

$$\nabla \times \bar{B} = \mu\bar{i} \qquad \text{(Eq. A5)}$$

$$\bar{B} = \nabla \times \bar{A}$$

$$\nabla \times \bar{B} = \nabla \times \nabla \times \bar{A} = (\nabla \cdot \bar{A}) - \nabla^2 \bar{A}$$

$$= -\nabla^2 \bar{A}$$

$$\nabla \times \bar{B} = \mu\bar{i} = -\nabla^2 \bar{A} \qquad \text{(Eq. A6)}$$

$$\nabla^2 \bar{A} = -\mu\bar{i}$$

Eliminating $\bar{B}$ from Equation A5,

$$\frac{d}{dt}\nabla \times \bar{B} = \frac{d}{dt} \mu\bar{i}$$

$$\nabla \times \frac{d}{dt} \bar{B} = \mu \frac{d\bar{i}}{dt}$$

$$\mu\frac{d}{dt} \bar{i} = - \nabla \times (\rho \nabla \times \bar{i})$$

$$\frac{\mu}{\rho} \frac{d\bar{i}}{dt} = \nabla^2 \bar{i} - \nabla (\nabla \cdot \bar{i}) = \nabla^2 \bar{i} \qquad \text{(Eq. A7)}$$

Eliminating $\bar{i}$ from Equations A4 and A6,

$$\bar{i} = - \frac{1}{\rho} \frac{d\bar{A}}{dt} = - \frac{1}{\mu} \nabla^2 \bar{A}$$

$$\frac{\mu}{\rho} \frac{d\bar{A}}{dt} = \nabla^2 \bar{A} \qquad \text{(Eq. A8)}$$

Eliminating $\bar{i}$ from Equations A3 and A5,

$$\nabla \times \bar{i} = - \frac{1}{\rho} \frac{d\bar{B}}{dt} = \frac{1}{\mu} \nabla \times \nabla \times \bar{B}$$

$$\frac{\mu}{\rho} \frac{d\bar{B}}{dt} = \nabla^2 \bar{B} \qquad \text{(Eq. A9)}$$

From Equation A7,

$$\frac{\mu}{\rho} \frac{d\bar{i}}{dt} = \nabla^2 \bar{i}$$

if $\bar{i} = \vec{i}_z \exp(j\omega t) = \vec{i}_z$ a phasor

then

$$\frac{d\bar{i}}{dt} = j\omega \vec{i}_z$$

$$\frac{\mu}{\rho}\frac{d\bar{i}}{dt} = jp\vec{i}_z$$

where $p = \omega\mu\sigma$

$$\nabla^2\bar{i} = \frac{d^2\vec{i}_z}{dr^2} + \frac{1}{r}\frac{d\vec{i}_z}{dr} \qquad \text{(Eq. A10)}$$

Let $V = (jp)^{1/2}r$ then

$$jp\vec{i}_z = \frac{d^2\vec{i}_z}{dr^2} + \frac{1}{r}\frac{d\vec{i}_z}{dr} \qquad \text{(Eq. A11)}$$

becomes

$$\frac{d^2R}{dV^2} + \frac{1}{V}\frac{dR}{dV} - (1 + \frac{n^2}{V^2})R = 0 \qquad \text{(Eq. A12)}$$

where $n = 0$.

Equation A12 is now in the form of Bessels modified function of order

zero. The solution is:

$$\vec{i}_z = \vec{C}I_0(V) + \vec{D}K_0(V)$$
$$= \vec{C}I_0[(jp)^{1/2}r] + \vec{D}K_0[(jp)^{1/2}r] \qquad \text{(Eq. A13)}$$

Since the sample is a solid cylinder, $K_0 = \infty$ when $x = 0$ and therefore

$\vec{D} = 0$ and Equation A13 becomes:

$$\vec{i}_z = \vec{C}I_0[(jp)^{1/2}r]$$

Using the boundary conditions at $r = a$, $\vec{i}_z = i_0$:

$$\vec{C} = i_0/I_0[(jp)^{1/2}a] \qquad \text{(Eq. A14)}$$

$$\vec{i}_z = \{I_0[(jp)^{1/2}r]/I_0[(jp)^{1/2}a]\}\, i_0 \qquad \text{(Eq. A15)}$$

Since $B = \mu I/2\pi a$

from Equations A3 and A15 and dividing by $\exp(j\omega t)$:

$$\vec{I} = 2\pi a B/\mu = \frac{\{2\pi \rho (jp)^{1/2} a I_o'[\_jp)^{1/2} a]\}}{j\omega \mu I_o \ [(jp)^{1/2} a]} \ i_o \qquad \text{(Eq. A16)}$$

Replacing the electric field by the magnetic field and using the

Buckingham Pi Theory, Equation A16 becomes

$$\frac{\phi}{\pi a^2 B_1 \mu/\mu_o} = \frac{2 I_o'(j^{1/2} x)}{j^{1/2} x \ I_o(j^{1/2} x)} \qquad \text{(Eq. A17)}$$

where $x = \omega\mu\sigma a^2$

The relative change of flux, $\Delta\phi$, caused by eddy currents in the cylinder

is:

$$\frac{\Delta\phi}{\phi_o} = \frac{\phi - \pi a^2 B_1}{\pi a^2 B_1} = \frac{\mu}{\mu_o} \frac{2 I_o'(j^{1/2} x)}{j^{1/2} x I_o(j^{1/2} x)} - 1 \qquad \text{(Eq. A18)}$$

Using the polar form and separating into real and imaginary parts:

$$-\alpha \frac{L_1}{L_o} = \frac{\mu}{\mu_o} \frac{2M_1(x)}{xM_o(X)} \cos[\theta_1(x) - \theta_o(x) - 3\pi/4] - 1$$

$$-\alpha \frac{R_1}{\omega L_o} = \frac{\mu}{\mu_o} \frac{2M_1(X)}{xM_o(X)} \sin[\theta_1(X) - \theta_o(X) - 3\pi/4]$$

Footnotes

[1]W.R. Smythe, _Static and Dynamic Electricity_ (New York, McGraw-Hill Book Company, 1950), 2nd ed., p. 395.

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Glossary

Accelerator: A chemical used to speed up a reaction or cure.

Adhesion: The state in which two surfaces are held together by interfacial forces.

Adhesive: A substance capable of holding materials together by surface attachment.

Aging: Subjection of a material to temperatures of 150°C for 1000 Hours.

Ambient Temperature: The temperature of the surrounding environment.

Anhydrides: Products which result from the removal of water from organic acids.

Arc Resistance: The time required for an arc to establish a conductive path in a material.

Arc Track: See arc resistance.

Bond: The union of materials by adhesives.

Bond strength: The unit load applied in mechanical properties required to break the assembly in or near the place of bond.

Catalyst: See hardener.

Cross-linking: Chemical links that are set up between molecular chains.

Cure: To change the chemical properties of a material by chemical reaction.

Curing Temperature: The temperature at which the material is subjected to cure.

Curing Time: The time it takes for a material to be properly cured.

Encapsulate: To coat an assembly with a thixotropic coating.

Epoxy: Thermosetting polymers containing an oxirane group.

Filler: An inert material added to adhesives to modify physical properties.

Hardener: A chemical added to a resin to cause curing or hardening.

Heat Distortion: The temperature at which a standard list bar deflects a stated amount under a stated load.

Hydroxyl Group: A chemical group consisting of one hydrogen atom plus one oxygen atom.

Inhibitor: A chemical added to resins to slow down the curing reaction.

Molecular Weight: The sum of the atomic masses of the elements forming the molecule.

Polyimide: High temperature resins made by reacting aromatic dianhydrides with aromatic diamines.

Polymer: A compound formed by the reaction of simple molecules.

Pot Life: The time a resin is usuable as a liquid after catalysts, fillers, etc. are added.

Resin: High molecular weight organic material with no sharp melting point.

Shelf Life: The length of time an epoxy can be stored without losing any of its original properties.

Thermoplastics: Plastics capable of being repeatedly softened by increases in temperature and hardened by decreases in temperature.

Thermosets: Resins which cure by chemical reaction due to heat, and when cured, cannot be resoftened by heating.